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Automatic Interfacing System for Sampling Total Mercury in Stationary Source Emissions

Geomet, Inc, Pomona, CA

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Automatic Interfacing System for Sampling Total Mercury in Stationary Source Emissions

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16. ABSTRACT

An interfacing system to sample total mercury emissions in source streams and suitably condition, dilute, and transport the sample to a mercury measuring instrument was designed, fabricated, and tested. The system consists of three components: a conditioner, a diluter, and a pump module. The conditioner contains a furnace to thermally decompose compounds at temperatures up to 1000°C and a liquid scrubbing system to remove particulates and interfering gases, such as sulfur dioxide and nitrogen dioxide. The diluter module is used at sources where mercury levels are above the calibration range of the measuring instrument. The pump module draws the sample through the system, exhausts the waste scrubber liquid, and maintains a constant pressure in the analyzer.

Field tests were conducted at a coal-fired power plant. Good correlation was obtained between the interface/photomatic analyzer and a reference manual procedure. Instrumental results ranged from 1.74 to 6.96 $\mu g/m^3$, with mean value of 4.23 $\mu g/m^3$; reference method results varied from 1.60 to 7.25 $\mu g/m^3$, with a mean value of 4.66 $\mu g/m^3$.

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by

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ABSTRACT

This program was initiated with the objective of developing an automatic instrumental interface which will permit the determination of low levels of mercury vapor while simultaneously removing relatively high levels of interferences from stationary emissions.

The system as designed, fabricated and tested included sample conditioner, dilutor and pump modules. The conditioner decomposes mercury compounds and scrubs out particulates and interfering gases. The diluter adds cleaned air to the sample to adjust elemental mercury vapor concentrations to levels which can be monitored with a DuPont Photometric Analyzer. The pump module draws sample through the system and maintains a constant gas pressure in the photometer.

Laboratory tests under conditions of high contamination confirmed the utility of the system. Utilizing resuspended fly ash at levels from 8.7 to 15.4 g/m^3 and 220 to 2175 PPM SO_2, replicate tests were conducted in the laboratory at mercury levels ranging from 0.038 to 1.70 mg/m^3. Removal of particulates and SO_2 was quantitative. No loss of mercury vapor was detected.

Field tests were conducted at Unit No. 2 of the John E. Amos Power Generation Plant of American Electric Service Corporation at St. Albans, W. Va., a coal-fired steam generating plant. The sampling probe assembly was installed through a port between the electrostatic precipitator and the 900 foot (high) stack in a duct (6.1 x 61 meters). Sample drawn at 22 lpm through a glass lined, SS probe were divided between bubblers and the interface-photometer system. In the majority of comparative tests run, the bubbler and instrumental procedures agreed very well in determination of the amounts of mercury in the exhaust. Values ranged from 1.7 to 7.0 $\mu g/m^3$ by the instrumental system; the range was 1.7 to 7.3 $\mu g/m^3$ by the bubbler method. The detection limit was 0.4-0.5 ug/m^3. Performance by the instrumental system was stable and reliable.

This report was submitted in fulfillment of Contract No. 68-02-1789 by Geomet, Incorporated under the sponsorship of the U.S. Environmental Protection Agency. The report covers the period from 5 May 1975 to 30 April 1976.

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We also wish to acknowledge the assistance rendered by the American Electric Power Service Corporation in permitting us to test our instrumentation at the J.E. Amos Power Generation Plant in St. Albans, W. Va. In particular, the assistance of Mr. T.T. Frankenberg and the plant management is gratefully noted.

SECTION 1

INTRODUCTION

The need for techniques to measure relatively low levels of mercury vapor in stationary source emissions presents a unique problem in removal of potential interferences from sample streams. Generally, each of the interferences is present at concentrations which may be as high as one million times that of the mercury vapor. An example of an industrial exhaust gas contained: 1-6 $\mu g/m^3$ of mercury in the presence of 0.5-1.0 g/m^3 particulates, approximately 1.0-1.6 g/m^3 fo sulfur dioxide and up to 1.1 g/m^3 of oxides of nitrogen (as NO_2). In order to measure the mercury vapor concentration by a photometric technique, the levels of these potential interferences must be reduced to low values.

The technical objective of this project was to develop a relatively sophisticated automatic interfacing system which will sample total mercury in source streams and suitably condition, dilute and transport the sample to a mercury measuring instrument.

Subtasks in development of the interface system included:

1. Design and construction of interface instrumentation so that a representative total mercury sample will be obtained from the source stream. This system shall provide for conditioning the sample and for dynamic dilution to reduce high mercury concentrations which are encountered at some sources to levels within the calibration range of the measuring instrument. Conditioning includes the conversion of all mercury compounds to an elemental state and the reduction of gaseous and particulate interferences to levels which do not interfere with the photometric procedure.
2. Test of the system in the laboratory and the field. The field test shall be conducted utilizing a source having high particulate loading and high sulfur dioxide concentrations.
3. Evaluation and interpretation of laboratory and field data including estimation of the following factors: accuracy, precision, sensitivity, stability, response time, interferences and reliability.
4. Delivery of the interface system to EPA at the end of the study together with complete operating procedures.

SECTION 2

CONCLUSIONS

An interface system consisting of three main modules has been designed, fabricated and tested. It consists of a conditioner, a diluter and a pump module. These components may be assembled into several sample processing configurations depending on the requirements of the source sample. All testing of the interface in the laboratory and the field was carried out in conjunction with a DuPont 400 Photometric Analyzer.

The conditioner contains two main functions, a furnace for heating the gases and a liquid scrubber. The furnace is designed so that mercury compounds may be thermally decomposed at temperatures to 1000°C, if required. It is usually operated at 300-400°C. The unit also contains a liquid scrubbing system for removal of particulates and interfering gases such as sulfur dioxide and nitrogen dioxide. The scrubber utilizes concurrent processing of the sample gas flowing at 2-2.5 liters/minute with solutions of sodium bicarbonate or sodium and ammonium bicarbonate usually pumped at the rate of 5 ml/min. through a twelve foot coil. This procedure removes all measurable traces of SO_2 and NO_2 and >97% of particulates even at particle loadings in the range of 10-12 g/m^3. At very high particle loadings a heated prefilter may also be added. These three functions may be used separately or in combination.

The diluter module which regulates sample flow to the DuPont Photometer can be operated in two modes: (1) to dilute the sample with clean air by any ratio from 1/5000 to 1/19, or (2) without any dilution. Gas flow regulation is accomplished by use of two Hastings mass flowmeters. One unit controls the total flow at rates to 5 liters per minute. The second establishes the fraction of the sample gas which will be analyzed.

The pump module draws the sample gas through the system, exhausts the waste liquid into a reservoir from the scrubber and maintains a constant pressure in the photometric analyzer. This latter feature is required for reproducible measurements.

Laboratory testing of the full interface system was carried out at particulate levels ranging from 8.7-15.4 g/m^3 of fly-ash and at 220 PPM sulfur dioxide. This level of fly-ash necessitated the use of a heated prefilter to achieve complete removal of the interference from particulates. The furnace was operated at 400°C in these tests; the scrubber utilized 0.2M $NaHCO_3$ circulated at a rate of 5 ml/minute. Tests were replicated at mercury concentrations varying from 0.038 to 1.70 mg/m^3.

These tests demonstrated a number of operating features of the interface system:

(1) Particulates at high concentrations can be reduced to undetectable limits by the prefilter and scrubber components. The scrubber alone can remove >97% of particulates even at very high loadings. By removing a major portion of the particulates in a prefilter, the scrubber-prefilter system achieves 100% efficiency.

(2) Sulfur dioxide was removed with nearly 100% efficiency by the scrubber.

(3) Mercury losses were undetectable.

In tests with dimethyl mercury and with the furnace operating at 400°C the system quantitatively yielded elemental mercury. No mercury losses were detected.

Tests of the diluter operating with dilution ratios between 1/21 and 1/110 showed that dilutions can be controlled readily by use of the two flowmeter control console. At the lower dilution ratios, experimental measurements of diluted concentrations agreed with the values set on the controls within ± 2%; at the highest dilutions the agreement was ± 4%.

Field tests of the interface system were carried out at Unit No. 2 of the J.E. Amos Power Generation Plant of the American Electric Service Corporation at St. Albans, W. Va. The test system was positioned on top of a duct between the electrostatic precipitators and the 900 foot stack. Operating conditions within the duct were: (1) Temperature, 280-320°F; (2) Air Velocity, 26.8-35.8 m/sec; (3) Gas Flow Rate, 1000-1300 m^3/sec; (4) The estimated exhaust composition was NO_x ~600 PPM, SO_2 400-600 PPM, particulates 0.5-1.0 g/m^3.

A heated, six-foot, glass lined, stainless steel probe was extended 41.9 cm into the duct at a position 1.9 meters from the duct wall. Gas was drawn from the duct at 22 liters/minute but only 2.5 liters/minute was passed to the instrumentation. A zero baseline for the system was established by passing air through a small bed of silver-alumina tablets. Analyses were obtained utilizing the full interface system in conjunction with the DuPont Photometric Analyzer operating at the 253.7 nm wave length and by use of the conventional bubbler collectors followed by analysis by the standard reduction procedure. An end-to-end calibration procedure was also employed. The two sets of data were compared in 37 sets of measurements.

A good correlation between the instrumented analyses obtained by use of the interface and the DuPont Photometric Analyzer and the reference manual procedure was obtained. Concentrations of mercury vapor in the exhaust gases were low. Instrumented results ranged from 1.74 to 6.96 ug/m^3 with a mean value of 4.23 ug/m^3. Reference method results varied from 1.60 to 7.25 ug/m^3 with a mean result of 4.66 ug/m^3. These results demonstrate that the interface system rejects the interfering exhaust components adequately allowing mercury vapor to pass through the system for photometric analysis.

SECTION 3

RECOMMENDATIONS

The automated interface system has been fabricated and tested in a variety of configurations. Its performance has been proven under relatively stringent field conditions. However, some beneficial modifications may be visualized.

The primary area in which improvements seem required is related to field calibration of the system during monitoring operations. For example, during the tests at the J.E. Amos Power Generation Plant a method for end-to-end configuration of the system calibration was established. The procedure was relatively time-consuming to carry out and required close attention from the operator. In essence, the procedure required (1) establishment of a clean air baseline signal, (2) introduction of a volumetric charge of mercury vapor by syringe immediately behind the probe and (3) determination of system response by the instrumental and bubbler procedures. As part of this procedure the three way valve shown in Figure 1 , page 5 is adjusted to draw ambient air through the silver-alumina tablet bed and thence through the photometer to establish a baseline response. In continuous monitoring operations, this procedure might be automatically controlled and programmed to take place for a short interval, such as five minutes, during every two hours. This would confirm the consistent performance of all components of the monitoring system.

An easily manipulated reference mercury source which can be handled in awkward physical situations would be highly desirable. Preferably, the source should operate to introduce a precalibrated mercury vapor sample into the inlet to the interface. This calibration standard would remove the necessity for time-consuming measurements utilizing the reference* bubbler collection technique. Its introduction might also be programmed and automated.

For operations in which sulfur trioxide is present, it would be desirable to establish a technique for easy replacement of the filter placed immediately downstream from the conditioner. This filter removes sulfuric acid mist aerosol which otherwise interferes with the operation of the Photometric Analyzer. A housing containing more than a single filter is needed so that sample gas streams may be readily switched between in-place filters. This change might also be automated in continuous operations.

These improvements would complement the system in a wide variety of monitoring applications.

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* Federal Register, Volume 38 (No. 66), pp. 8820-8850, April 6, 1973

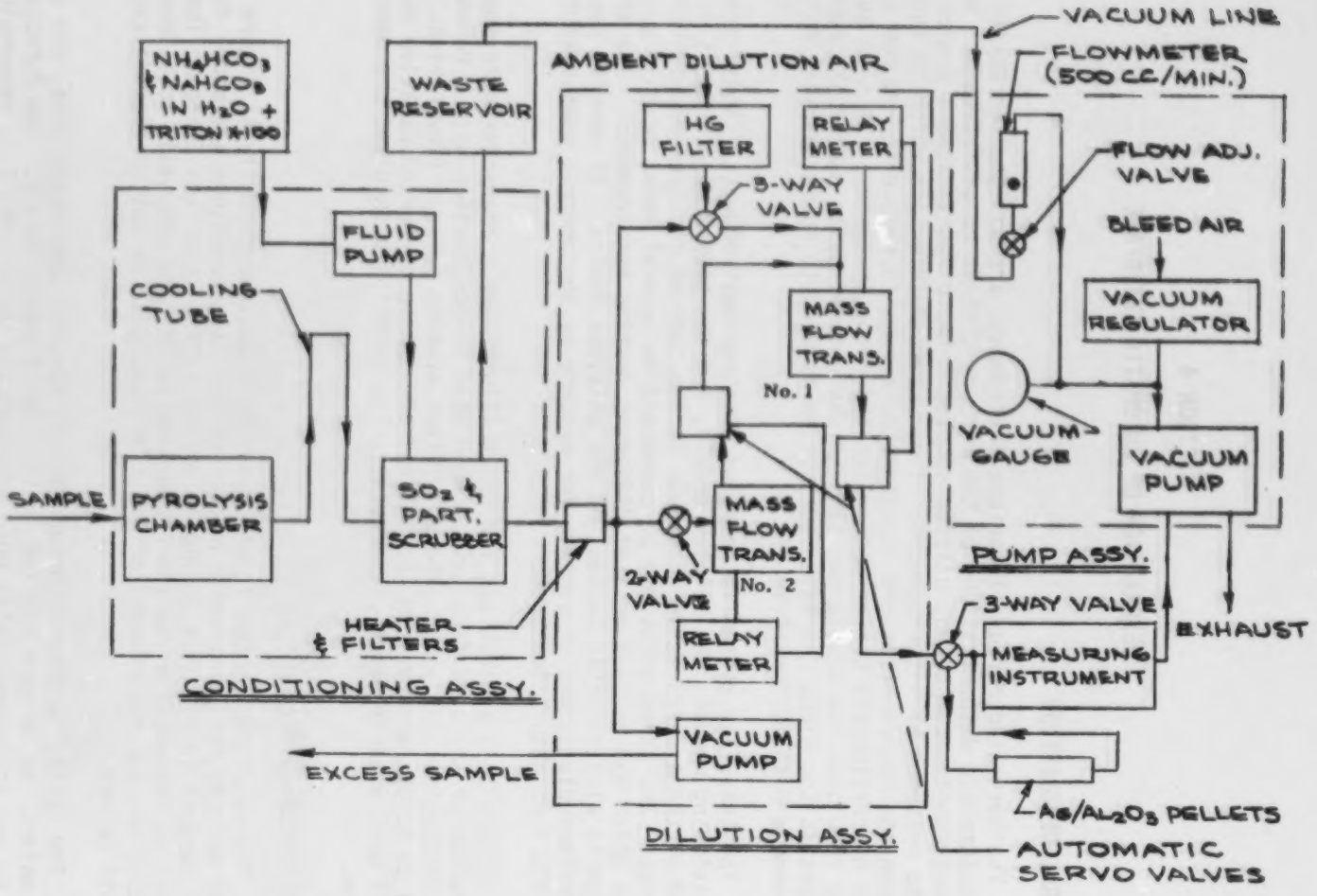


Figure 1. Interface System showing components of modules.

SECTION 4

DESCRIPTION OF AUTOMATIC INTERFACE

GENERAL DESCRIPTION

The Automated Mercury Interface instruments are designed to assist spectrophotometric determinations of the mercury content of gas streams. Normally, a spectrophotometer cannot utilize samples taken directly from a source because of the presence of interferences. The Automated Interface which is interposed between the source probe and a photometer serves to remove the common interferences of particulate matter and sulfur and nitrogen oxides. It also automatically regulates the sample gas flow to the optical cell, and if the mercury content is too high, the interface system is used to quantitatively dilute the sample with mercury-free air.

The interface system is composed of three main modules. These are the conditioning module, the diluter module, and the pump module. As it is composed of three units it is adaptable to the testing requirements of various sources. The three units may be connected in several modes or only two of the three units may be used to meet different testing requirements. The Interface System is also portable because of its unitized design. It permits testing of a considerable number of sources for mercury by photometric measurements, at the 253.7 nm absorption peak.

Figure 1 is a block diagram which illustrates a characteristic mode of operation of the interface system when used in conjunction with a DuPont 400 Photometric Analyzer. The 10% silver/alumina pellets (lower right) are used to scrub mercury from the ambient atmosphere or for comparative measurements when interferences are present. The principles involved are described below.

Conditioning Module

The conditioning module which is shown photographically in Figure 2 prepares the gas for photometric measurements. Its functions are shown in the left part of Figure 1, a schematic of the entire Interface System. The conditioner is responsible for the conversion of mercury compounds to elemental mercury and for the removal of interferences such as sulfur dioxide and particulate matter.

The initial process: After passing through the intake line, the sample gas enters the furnace located in the conditioning module. The furnace which may be set to automatically hold temperatures up to 1000°C is responsible for

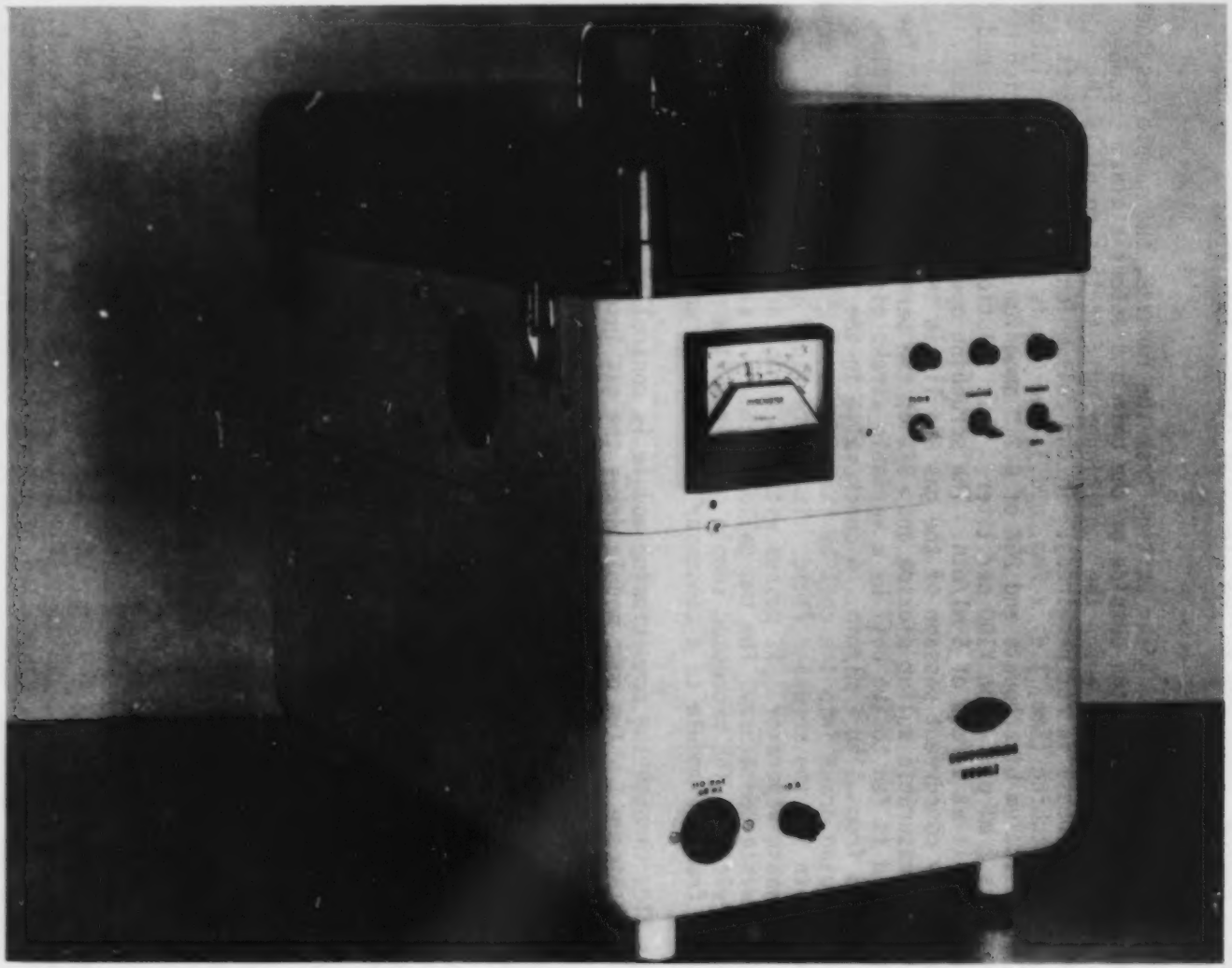


Figure 2. Conditioning Module

the decomposition of mercury compounds. This section is usually operated in the range 300-400°C. The sample is heated as it passes through a coil of 10 feet of stainless steel tubing (0.635 mm, i.d. x 0.953 mm, o.d.). This heating decomposes mercury compounds to the elemental state. This process may be bypassed if it is not needed.

The scrubber process: After exiting from the furnace, the sample gas goes through a cooling tube on the outside of the unit and enters a glass scrubber coil. (The gas sample may enter the conditioner unit at this point if the decomposition process is not required). In the coil which is made of 12 ft. of pyrex glass tubing (5mm, i.d. x 7mm, o.d.), the gas is mixed with a solution of 80% 0.2 M sodium bicarbonate and 20% of 0.2 M ammonium bicarbonate which also contains 2 ml of Triton X100 per liter. This solution is usually pumped through the coil at a rate of 5 ml/min. The scrubbing operation which is carried out by concurrent passage of the gas and liquid through the coil, removes all measurable sulfur dioxide and > 97% of the particulate matter. The scrubber fluid is fed to the coil by a variable stroke piston pump. The flow can be varied from ~1 to 7 ml/min. A check valve in the line prevents excess fluid from being drawn into the coil by the vacuum. A 2 gallon container serves as a reservoir for the scrubber fluid. The waste fluid is separated from the gas in the waste separator. It passes with 0.5 1/min. of the sample gas to a 2 gallon waste container. The two gallon containers allow a full 24 hours between servicing. The processed sample gas, at a flow rate of 2 liters/minute, exits from the module to a condenser where excess water is removed.

Gas flow through the conditioning module is controlled by the pump module as shown in Figure 1. The pump module draws the gas into the conditioning module at approximately 2.5 1/min.

In cases where mercury compounds are not of concern, the furnace may be bypassed. This is easily accomplished by connecting the sample source to the inlet to the scrubber section which is located on the right side of the module, or a 12/5 socket joint may be fitted to the sample line (or probe) and then connected by tubing to the inlet to the scrubber portion of the module. By this method, the unheated gas goes directly into the scrubber coil without passage through the furnace. This technique should be used in sampling operations where mercury compounds or vapors have been previously exposed to high temperatures.

The Diluter Module

The diluter module which is shown in Figure 3 automatically regulates gas flow to the photometer, and it may simultaneously be used to dilute high mercury concentrations to levels applicable to the calibration range of the analytical instrument. Gas flow regulation is accomplished using a 0.5 1/min. single set point Hastings Mass Flowmeter and an automatic valve which is controlled by the flowmeter. Any desired flow rate up to 5 1/min (± 1%) may be obtained by placing the set point of the total flowmeter at the desired level. The valve is automatically controlled to achieve the indicated total flow rate.

When dilution is required it is accomplished by using the lower flow range (0-100 ml/min.) Hastings Mass Flowmeter and a second automatic valve to divert

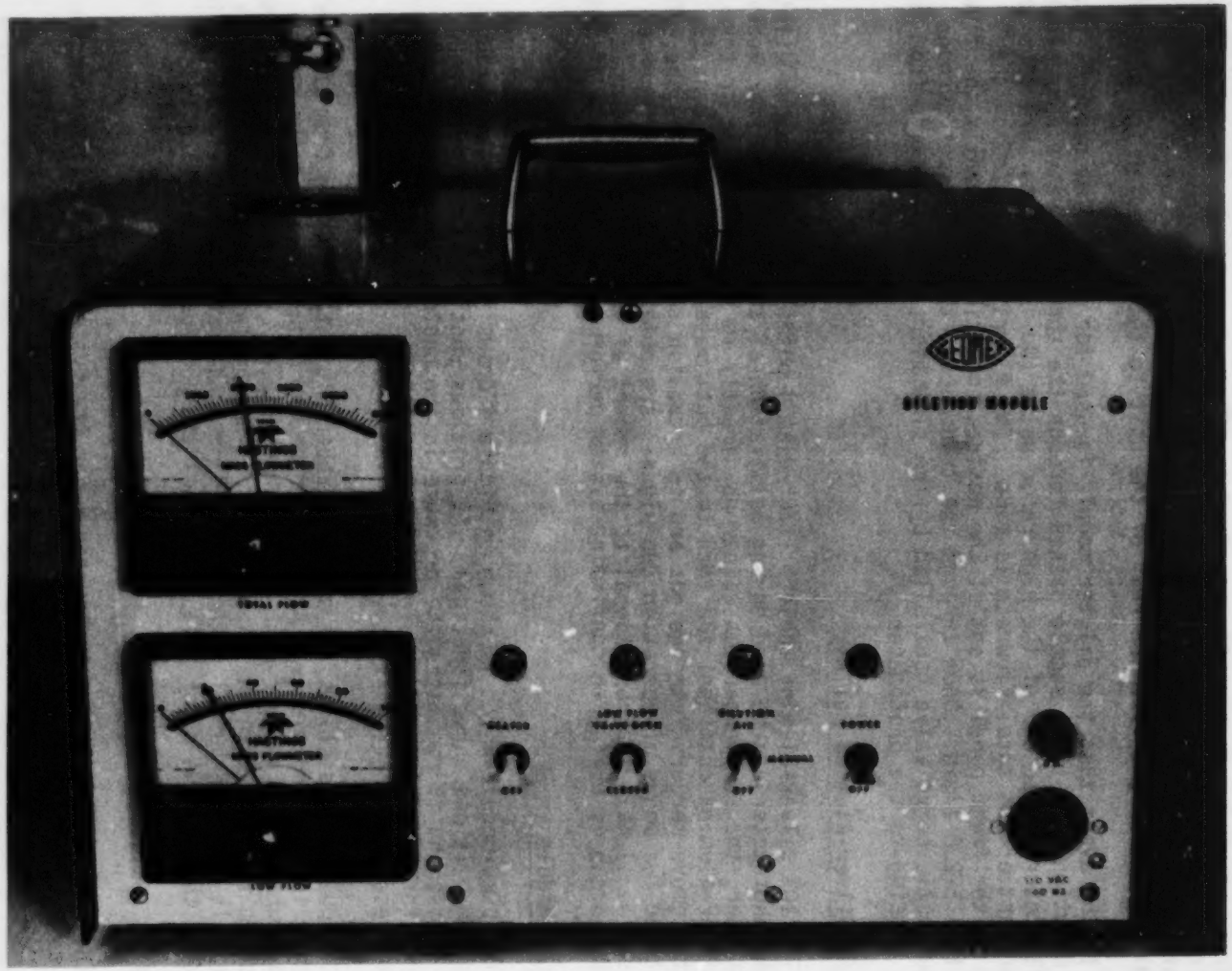


Figure 3. The Diluter Module

a portion of the processed sample stream into a clean air stream. The scheme is shown in Figure 1, center section. The indicator of the low flow range meter (Meter No. 2) is used to select the desired flow rate of the sample from the total stream. With this sensor in use, the two-way valve is open. The three-way valve is open to the air from the mercury filter, but closed to sample air. Excess sample is exhausted through the vacuum pump. In this operation dilutions from 1/5000 to 100/1900 may be obtained. After dilution, the gas is discharged from the rear of the unit to the photometer for analysis.

In the event that the diluter module is used for total flow control, rather than dilution, the total sample passes through the three-way valve and enters the flowmeter transducer of the high range valve (No. 1). It is here that the measurement of the total flowrate is made. The flowrate indicated by the meter on the front panel is set at the desired condition. Generally the flow rate is 1.5-2.0 1/min. Once through the transducer, the gas passes through the automatic servo valve controlled by the flowmeter, and then exits from the unit, passing to the photometer.

The 10% Silver-Alumina Pellets

The 10% silver-alumina tablets (1/8" diam.) positioned between the diluter and the photometer can be used during calibration of the system. After leaving the diluter, the sample gas can either go directly to the photometer or through the silver-alumina absorbent and then to the photometer. This absorbent is used to remove mercury from the gas and to establish the zero or baseline. By use of the two modes of operation two signals are obtained. The difference between the signals is proportional to the amount of mercury in the sample. This technique is useful in confirming system performance in a variety of tests.

The Pump Module

The pump module which is shown in Figure 4 draws the sample gas through the system. It has three functions. It provides suction to the sample and waste lines, and it regulates the pressure in the photometer cell. This is required for reproducible measurements. A magnehelic gauge indicates the vacuum present in the system.

The pump module allows the spent scrubber fluid to be drawn from the waste separator in the conditioner. A flowmeter on the module monitors the sample gas flow which is drawn off with the scrubber fluid. A Thomas air pump provides the vacuum for the waste and sample lines. The sample gas enters at the back of the unit from the DuPont photometer and from the waste container, and it is exhausted through the pump. Figure 1 shows the procedure schematically.

OPERATING INSTRUCTIONS

The detailed operating instructions for the system as established in the tests as described in Section 5 are included in Appendix A.

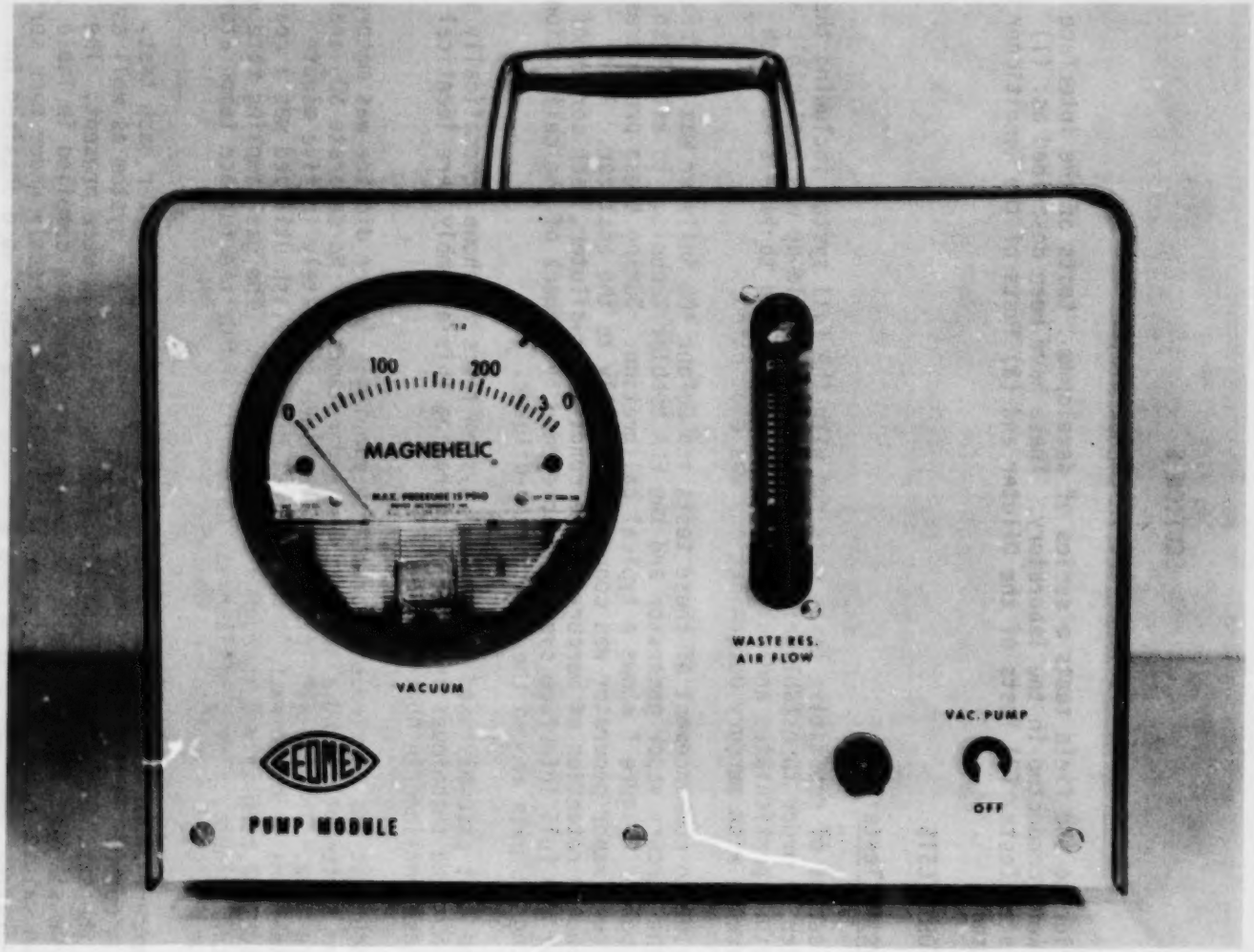


Figure 4. The Pump Module

SECTION 5

TESTS OF THE INTERFACE SYSTEM

Prior to the field tests a series of development tests of the interface system were conducted in the laboratory. These have been described as: (1) Full System Tests, (2) Tests of the Diluter and (3) Tests of the Conditioner Subsystem.

LABORATORY TESTS

Full System Tests

A series of repetitive tests were run with the full system including the prefilter in order to establish: (1) whether mercury hold-up was negligible and (2) whether particulates and sulfur dioxide were reduced to levels at which interference with mercury determinations was eleminated.

Prior to commencement of these tests the DuPont 400 Analyzer was calibrated using the mercury vapor generator and the EPA bubbler technique to establish its response. Figure 5 shows a typical calibration. During these procedures the mercury vapor generator was connected directly to the analyzer. As a check on the retention of mercury in the conditioner-diluter, each series of tests of the full interface system was preceded by a check of the calibration utilizing a bypass around the conditioner-diluter.

The test assembly used in the full system tests is shown schematically in Figure 6. The techniques involved in operating this assembly were identical with those outlined in Appendix A.

Each test was carried out for thirty minutes. Sulfur dioxide was metered into the system utilizing a needle valve and rotameter. No separate SO_2 analyses were made. The fly-ash level was determined separately in five minute determinations prior to each 30 minute test. The fly-ash utilized was a coal ash which had been seived through a 200 mesh screen. The gas sampling rate was 2.5 liters/minute. The prefilter was heated to 400°C; the furnace temperature was also 400°C.

Mercury vapor concentrations were measured before and after each test, during a period when fly-ash and SO_2 were not added to the system as well as during the test period when both potential interferences were present. The data from six sets of runs replicated five times each are compiled in Table 1. The mean values for each set of five tests are shown directly under each series of replicates. This table is a record of the data which were obtained for analyses of six levels of mercury in the presence of 11.8-12.1 g/m^3 (mean values) of fly-ash at an SO_2 concentration of 220 ppm. Mercury concentrations were

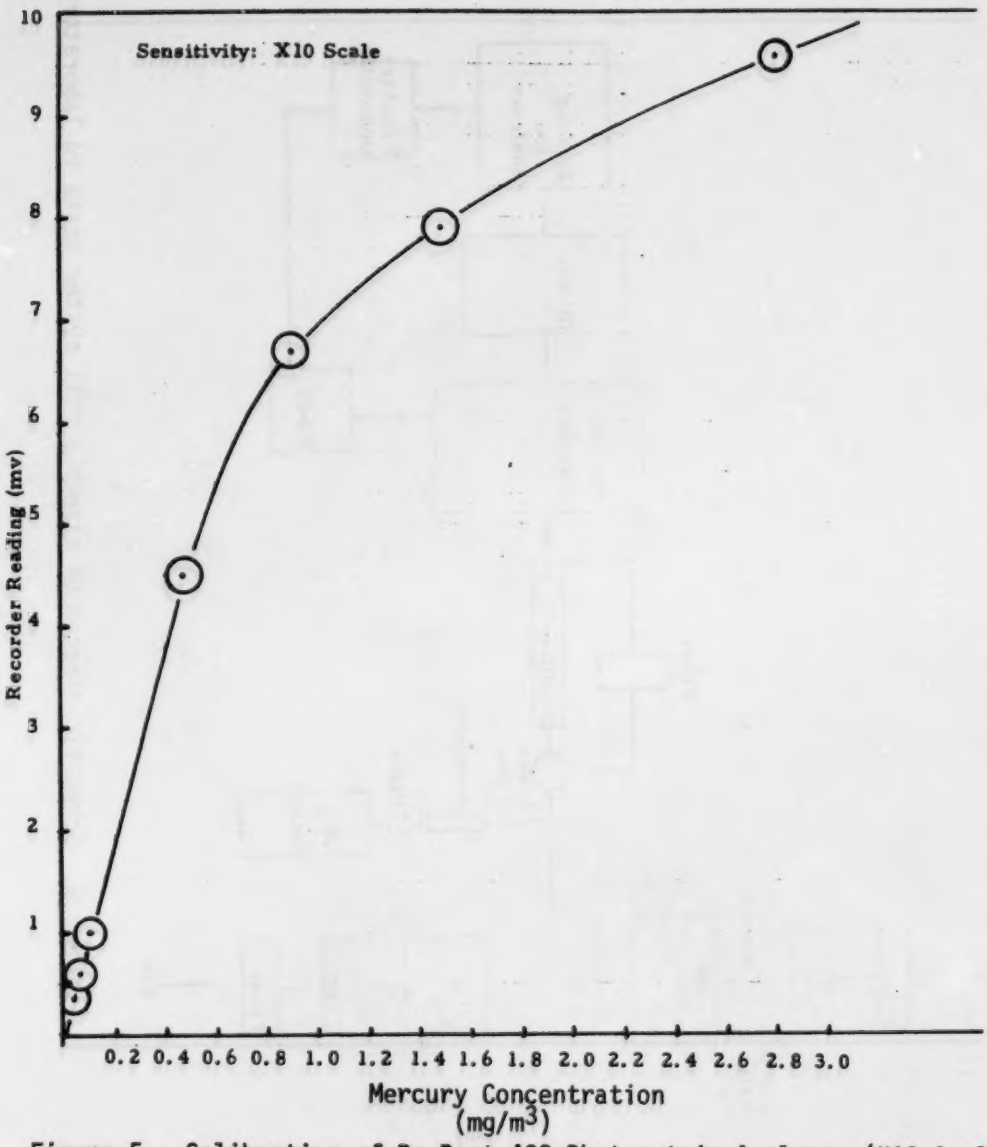


Figure 5. Calibration of Du Pont 400 Photometric Analyzer (X10 Scale).

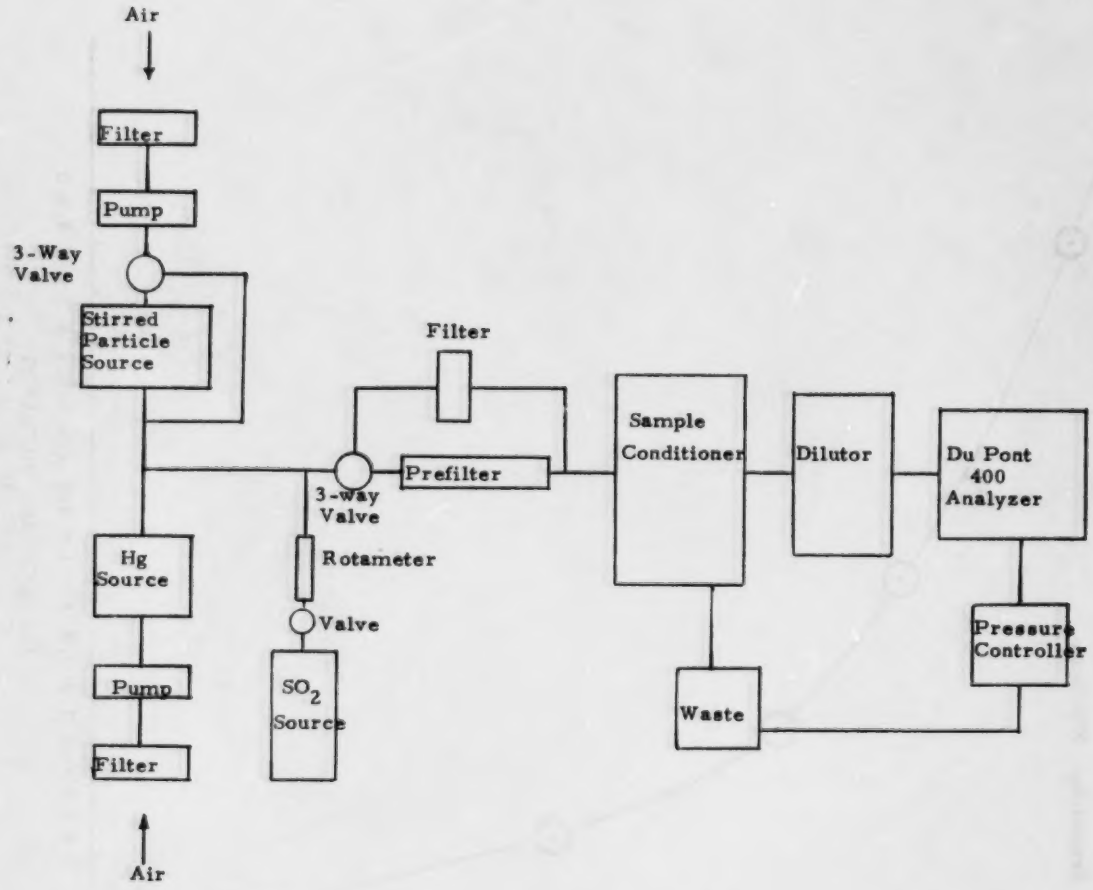


Figure 6. Schematic diagram of assembly full system tests in laboratory.

TABLE 1. FULL SYSTEM TESTS

Test No.	Mean Recorder Values (mv)			Mean Mercury Conc (mg/m^3)			% Hg Passed thru System	Particulate Level (g/m^3)	SO_2 Conc. (PPM)	System Pressure (cm H_2O)
	Before Test	During Test	After Test	Before Test	During Test	After Test				
1	0.90	0.93	0.90	0.088	0.090	0.088	102.3	10.6	220	808
2	0.85	0.83	0.83	0.082	0.080	0.080	98.8	10.1	220	808
3	0.83	0.83	0.83	0.080	0.080	0.080	100.0	12.9	220	808
4	0.87	0.85	0.87	0.085	0.082	0.085	96.5	11.7	220	808
5	0.80	0.83	0.80	0.078	0.080	0.078	102.6	13.8	220	808
Mean	0.85	0.85	0.35	0.083	0.082	0.082	100.0	11.8	220	808
6	3.60	3.70	3.70	0.362	0.376	0.376	101.9	12.2	220	808
7	3.55	3.60	3.55	0.358	0.362	0.358	101.1	13.3	220	808
8	3.85	3.80	3.70	0.395	0.385	0.376	99.7	13.2	220	808
9	3.80	3.70	3.65	0.385	0.376	0.370	99.6	11.1	220	808
10	3.95	3.95	3.95	0.403	0.403	0.403	100.0	10.5	220	808
Mean	3.75	3.75	3.71	0.381	0.380	0.377	100.5	12.1	220	808
11	6.80	6.90	6.90	0.880	0.905	0.905	100.3	11.3	220	808
12	7.05	7.10	7.05	0.960	0.975	0.960	101.6	15.4	220	808
13	7.15	7.10	7.05	0.995	0.975	0.960	99.7	12.7	220	808
14	7.20	7.20	7.30	1.015	1.015	1.055	98.1	13.6	220	808
15	6.90	6.95	6.90	0.905	0.925	0.905	102.2	8.7	220	808
Mean	7.02	7.05	7.04	0.951	0.959	0.957	100.4	12.3	220	808

(continued)

TABLE 1 (continued)

Test No.	Mean Recorder Values (mv)			Mean Mercury Conc (mg/m^3)			% Hg Passed thru System	Particulate Level (g/m^3)	SO_2 Conc. (PPM)	System Pressure (cm H_2O)
	Before Test	During Test	After Test	Before Test	During Test	After Test				
16*	3.6	3.5	3.5	0.038	0.037	0.037	98.7	11.6	220	808
17*	3.9	3.8	3.8	0.042	0.041	0.041	98.8	12.7	220	808
18*	3.7	3.7	3.7	0.040	0.040	0.040	100.0	12.1	220	808
19*	3.4	3.4	3.4	0.036	0.036	0.036	100.0	13.3	220	808
20*	3.6	3.6	3.6	0.038	0.038	0.038	100.0	11.9	220	808
Mean	3.6(4)	3.6(0)	3.6(0)	0.038(8)	0.038(4)	0.038(4)	99.5	12.3	220	808
21	2.1	2.1	2.0	0.23	0.23	0.22	102.2	13.1	220	808
22	2.0	2.0	2.0	0.22	0.22	0.22	100.0	12.3	220	808
23	2.3	2.3	2.3	0.25	0.25	0.25	100.0	13.4	220	808
24	2.3	2.3	2.3	0.25	0.25	0.25	100.0	11.6	220	808
25	2.2	2.2	2.1	0.24	0.24	0.23	102.1	10.5	220	808
Mean	2.1(8)	2.1(8)	2.1(4)	0.23(8)	0.23(8)	0.23(6)	100.9	12.2	220	808
26	8.3	8.3	8.3	1.76	1.76	1.76	100.0	10.9	220	808
27	8.2	8.1	8.1	1.68	1.60	1.60	97.6	11.8	220	808
28	8.5	8.6	8.5	1.87	1.94	1.87	103.7	13.1	220	808
29	8.0	8.0	8.0	1.52	1.52	1.52	100.0	12.2	220	808
30	8.2	8.2	8.2	1.68	1.68	1.68	100.0	12.5	220	808
Mean	8.2(4)	8.2(4)	8.2(2)	1.70	1.70	1.69	100.3	12.1	220	808

* Measurements on the X1 scale; all others on X10 scale of Du Pont 400 Photometric Analyzer

determined by use of calibrations of the DuPont 400 Photometer which were made without use of the interface instrumentation.

The data for the thirty tests of the full system have been composited and are shown in Figure 7. Each point represents a mean value obtained from five tests. The fly-ash which was determined for each test, had a mean value of 12.1 g/m^3 and a range from 8.7 to 15.4 g/m^3. The sulfur dioxide was maintained at 220 ppm; it was metered into the system by use of a rotameter.

Comparisons of the mercury levels obtained in tests with the complete system with calibrations made without use of the interface instrumentation show no significant differences. The curves are essentially identical. It may be concluded that the technical objectives of the project have been achieved, at least when tested under laboratory conditions: (1) High levels of particulates and sulfur dioxide have been reduced by the interface instruments to insignificant or zero levels; no interferences are obtained in the photometric analysis of mercury vapor. (2) There is no evidence of hold-up of mercury vapor by the interface instrumentation. (3) Within the precision of the measurements, the analyses of mercury vapor by the system are identical whether the original sample stream contains SO_2 and fly-ash or none of these potential interferences.

Tests with Organic Mercury--

A comparison of peak heights obtained by introducing determined amounts of dimethyl mercury and elemental mercury was used to establish the efficiency of the prefilter and conditioner in converting dimethyl mercury to elemental mercury.

With the furnace and prefilter* at 400°C, a calibration curve was obtained with elemental mercury which was introduced at the prefilter inlet by means of a syringe. These mercury samples collected by bubbler after passage through the system. A $KMnO_4/HNO_3$ bubbler system was employed. The contents of each bubbler was analyzed by reduction with 20% $SnCl_2$ in 6 N HCl and by determination of the resulting peak signal on a Perkin-Elmer atomic absorption spectrophotometer.

For determinations of dimethyl mercury (DMM), a standard solution of DMM in xylene was prepared volumetrically using a micropipet. Known amounts of this solution were pipetted into the entry to the prefilter where it rapidly evaporated. After correcting for the solvent blank, amounts of elemental mercury obtained in the bubblers were determined.

The following data compare the experimentally determined amounts of mercury with the calculated amounts introduced volumetrically as DMM in xylene.

* Prefilter contains 60 g of pyrex wool.

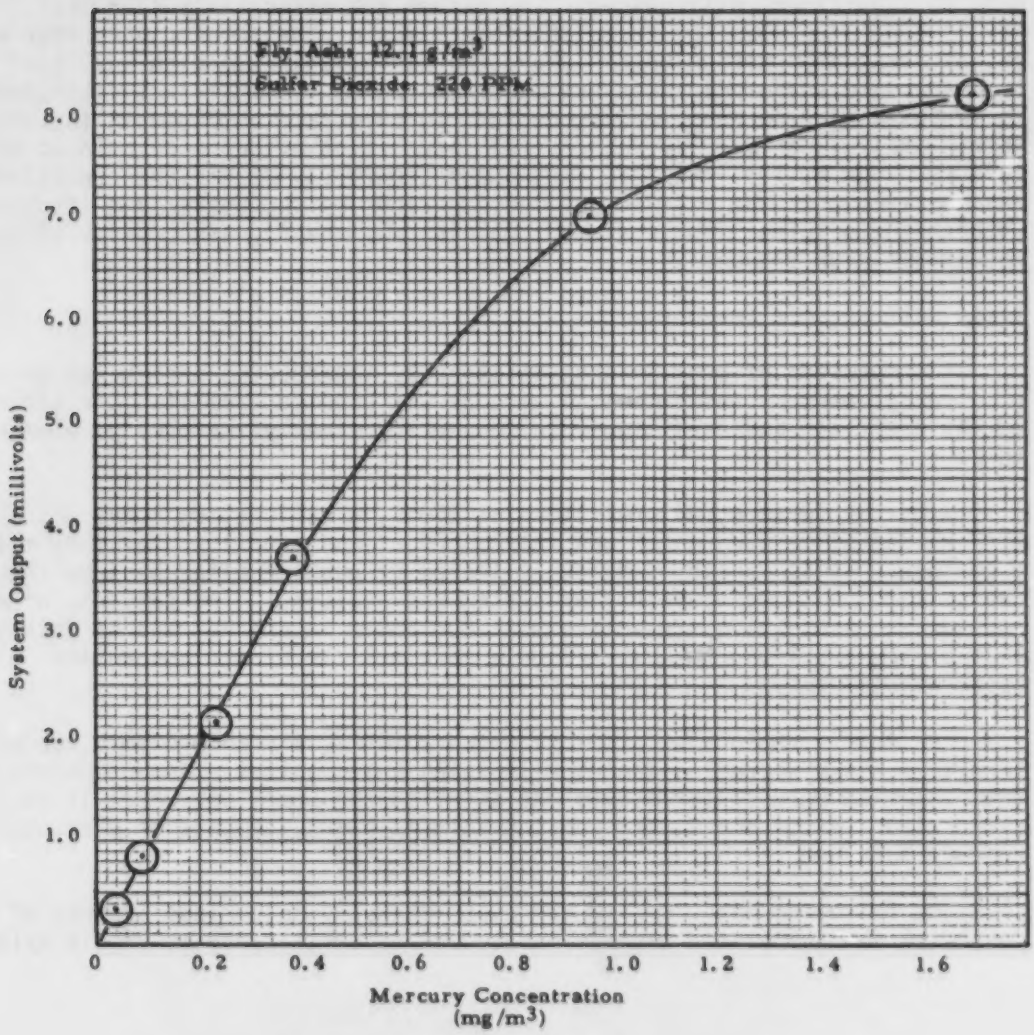


Figure 7. Full System Performance.
Mercury Analysis in presence of Fly-Ash and Sulfur Dioxide

Amount of Hg introduced as $(CH_3)_2$ Hg	Experimental Determination of Hg after Conversion
21 nanograms	21 nanograms
21 "	24 "
21 "	24 "
54 "	52 "
54 "	56 "
54 "	61 "
107 "	102 "
107 "	107 "
107 "	109 "

Each bubbler contained 0.5 ml of 0.25 $KMnO_4$, 2.5 nl of 1:1 HNO_3 and sufficient distilled water to make the volume up to 20 ml. Air flow was 2.5 l/min.

The data demonstrate that mercury introduced as dimethyl mercury is fully recovered in the $KMnO_4/HNO_3$ bubbler. Conversion of DMM to elemental mercury appears complete. No loss of mercury in either form was noted.

Response Times--

Response time times were run by connecting and disconnecting the mercury source stream immediately ahead of the pre-filter and determining the time to response and clean-up which resulted. The average responses were as follows:

Time to Initial Response - 10 ± 5 seconds

Time to Peak Response - 50 ± 9 seconds

Time to 90% Response - 46 ± 6 seconds

Time to Clean-up from Peak Response (Fall Time) - 66 ± 6 seconds

Time to 90% Clean-up - 42 ± 5 seconds

Detection Limits of System--

Expermimental tests of the detection limit of the entire system were run utilizing the mercury vapor generator. Initially, a steady low level output of mercury was established with the generator. By adjusting the physical parameters controlling the generator the signal was reduced until it was just

detectable on the X1 scale of the DuPont 400 Photometer. This is equivalent to 1/5 of a chart division on the recorder. For confirmation, the source was disconnected from the prefilter allowing the signal to subside to the zero baseline. Reconnection of the source established the detectability of the low level mercury concentration. This mercury level was then determined by use of the $KMnO_4/HNO_3$ bubbler. Replicate determinations of the detectable limit on the X1 scale show 0.4 ug/m^3 as the experimental limit. On the X10 scale of the photometer 4 ug/m^3 was detectable.

Precision--

The precision of mercury vapor determinations may be estimated on the basis of the standard deviations for each set of data included in Table 1. Summarized these are:

Test Nos.	Mean Mercury Conc. (mg/m^3)	Std. Deviation (mg/m^3)	C.V. (%)
16-20	0.038(4)	0.002(1)	5.5
1-5	0.082(4)	0.004(3)	5.2
21-25	0.23(8)	0.01(3)	5.5
6-10	0.380(4)	0.015(1)	4.0
11-15	0.959(0)	0.043(9)	4.6
26-30	1.70	0.16(1)	9.5
		Mean C.V.	5.7%

Thus, the estimated mean coefficient of variation (C.V.) for the six sets of data is 5.7%.

Accuracy, Stability and Reliability--

The system calibration is based on comparison of the DuPont Analyzer signals with standard bubbler determinations of mercury vapor, as quantitated by the Hatch and Ott procedure. Accuracy estimates are therefore based on the test values (Column 6 in Table 1) as compared with the mean values obtained from the determinations before and after each test as tabulated in Columns 5 and 7. These latter values were determined by use of the by-pass around the scrubber-diluter hardware.

For all thirty tests, determinations utilizing the full system agreed with those obtained by use of the by-pass within ± 1.6%. That is, recovery of mercury utilizing the interface was 100.3 ± 1.6% of that obtained without its use. Since these data were obtained during a period of three weeks, the stability and reliability of the interface system appears satisfactory.

Tests of the Diluter

A series of tests of the diluter were made with system inputs identical to the full system tests. That is, input particulate levels were 10-14 g/m^3 and the sulfur dioxide concentration was maintained at 220 ppm. Mercury vapor levels were varied from 0.94 to 1.87 mg/m^3. A series of dilutions ranging from 1/21 to 1/101 were tested. The data are shown in Table 2.

Comparison of the four right hand columns of Table 2 shows that the diluter yields the calculated concentrations of mercury vapor within a precision varying from ± 2 to ± 4%. Comparison of the measured mercury concentrations in Column 6 with the calculated concentrations in Column 8 shows good agreement. Calculated concentrations are obtained from input concentrations by application of the dilution factor obtained from the diluter settings. Measured output concentrations are obtained from the recorder reading in millivolts (Column 5) by use of a standard calibration.

It may be concluded that the diluter performs successfully and does not serve to trap mercury vapor.

Tests of the Conditioner Subsystem

Two series of tests were utilized to determine the preferred configuration of the conditioner: (1) the standard module consisting of the thermal converter and scrubber or (2) the same device to which a prefilter was added. At high particle loadings, the latter configuration improves the operating performance. Either modification may be used, depending on the properties of the source under test.

Tests of the Standard Module--

The sample conditioner module was tested with coal fly-ash utilizing the assembly shown schematically in Figure 6. The fly-ash was prepared by burning commerically-available anthracite (10.2% solids as ash) and sieving the ash through 200 mesh screens. About 250g of the sieved ash was placed in a two gallon carboy where it was agitated with a 2 inch propeller rotating at 3500 rpm. Air drawn from the top of the carboy at 2-2.5 lpm contained 5-15 g/m^3 of fly-ash. Although these concentrations are higher than the average exhaust of most power plants, they serve as an adequate test condition in the laboratory.

Table 3 shows the results of a series of fly-ash removal efficiency tests. The weight of ash collected on Filter No. 1 (Figure 9) during short periods of operation with the 3-way valve diverting the stream through the filter, served to quantitate the source level of particulates in the gas. The residue deposited on Filter No. 2 established values for particulates which are not removed by the process.

The procedure for determination of the ash on a filter included: (1) Each Gelman Versapore filter was weighed and inserted in a 1 inch (diameter) holder. (2) After completion of the test, the filters were dried for 20 min. at 110° C and reweighed. The tare weight of clean filters was ~0.060g.

TABLE 2. TEST OF DILUTER

Mercury Input Concentration		Dilution Volume Factor		Output			
				Measurements		Calculated	
Mean Reading (mvX10)	Conc. (mg/m^3)	Vol Sample / Vol Total		Mean Reading (mv,X1)	Conc. (mg/m^3)	Mean Reading (mv,X1)	Conc (mg/m^3)
6.8	0.94	100/2100	0.0476	4.3	0.045	4.3	0.0447
6.8	0.94	"	"	4.4	0.046	4.3	0.0447
7.0	1.03	"	"	4.6	0.049	4.6	0.0490
7.1	1.07	"	"	4.9	0.051	4.8	0.0509
8.0	1.54	"	"	7.2	0.074(5)	7.0(5)	0.0733
8.1	1.60	"	"	7.7	0.079	7.4	0.0762
8.2	1.67	"	"	7.8	0.080	7.9	0.0795
8.3	1.74	"	"	8.2	0.085	8.0	0.0828
8.5	1.87	"	"	8.6	0.089	8.6	0.0890
6.8	0.94	80/2080	0.0385	3.5	0.036	3.4(5)	0.0362
6.8	0.94	"	"	3.4	0.036	3.4(5)	0.0362
7.1	1.07	"	"	3.9	0.041	3.9(5)	0.0412
8.0	1.54	"	"	5.7	0.059	5.7	0.0593
8.1	1.60	"	"	5.9	0.061	5.9(5)	0.0616
8.2	1.67	"	"	6.2	0.064	6.2	0.0643
8.3	1.74	"	"	6.6	0.068(5)	6.4(5)	0.0670
8.5	1.87	"	"	6.8	0.070(5)	6.9	0.0720
6.8	0.94	60/2060	0.0291	2.6	0.027	2.6	0.0274
6.8	0.94	"	"	2.7	0.028	2.6	0.0274
7.1	1.07	"	"	3.0	0.031	3.0	0.0311
8.3	1.74	"	"	5.0	0.052	4.9	0.0506

(continued)

TABLE 2 (continued)

Mercury Input Concentration		Dilution Volume Factor		Output			
				Measurements		Calculated	
Mean Reading (mvX10)	Conc. (mg/m^3)	Vol Sample / Vol Total		Mean Reading (mv, X1)	Conc. (mg/m^3)	Mean Reading (mv, X1)	Conc. (mg/m^3)
8.0	1.54	50/2050	0.0244	3.8	0.039(5)	3.6(5)	0.0376
8.1	1.60	"	"	3.8	0.039(5)	3.8	0.0390
8.2	1.67	"	"	4.0	0.041(5)	3.9(5)	0.0407
8.3	1.74	"	"	4.2	0.044	4.1	0.0425
8.5	1.87	"	"	4.4	0.046	4.4	0.0456
6.8	0.94	40/2040	0.0196	1.8	0.019	1.8	0.0184
6.8	0.94	"	"	1.8	0.019	1.8	0.0184
7.1	1.07	"	"	1.9	0.020	2.0	0.0210
8.0	1.54	20/2020	0.00990	1.5	0.016	1.4	0.0152
8.1	1.60	"	"	1.5	0.016	1.5	0.0158
8.2	1.67	"	"	1.5	0.016	1.5(5)	0.0165
8.3	1.74	"	"	1.7	0.018	1.6(5)	0.0172
8.5	1.87	"	"	1.9	0.020	1.7(5)	0.0185

TABLE 3. PARTICULATE COLLECTION

Test No.	Collection Time @ 2 l/m		Ash Collection (g)		Concentration of particulates (g/m^3)		Scrubber Efficiency (%)	Scrubber Operation
	No. 1 Filter	No. 2 Filter	No. 1 Filter	No. 2 Filter	Inlet	Exhaust		
1	3 min	9 min	0.0454g	0.0037g	$7.6 g/m^3$	$0.21 g/m^3$	97.3%	4.4 ml/min of 0.2M $NaHCO_3$ containing 1 ml/l of 10% Triton X100
2	4	15	0.0419	0.0049	5.2	0.16	97.0	5.2 ml/min
3	3	15	0.0588	0.0051	9.8	0.34	96.9	6.0 ml/min
4	4	15	0.0973	0.0066	12.2	0.66	94.9	6.0 ml/min
5	3	15	0.0307	0.0070	5.1	0.23	95.7	6.5 ml/min
6	3	15	0.0341	0.0072	5.7	0.24	96.0	8.5 ml/min
7	2	18.75	0.0285	0.0053	7.1	0.14	98.1	6.5 ml/min
8	3	141	0.0791	0.0747	13.2	0.26	98.1	6.5 ml/min
9	3	15	0.0542	0.0111	9.0	0.37	96.1	8.5 ml/min
10	3	12	0.0951	0.0061	15.9	0.25	98.5	8.5 ml/min 0.2M $NaHCO_3$ + 3.3 ml/l Triton X-100
11	3	15	0.0437	0.0047	7.3	0.16	97.9	8.5 ml/min 0.2M $NaHCO_3$ + 7.3 ml/l Triton X-100
12	3	30	0.0285	0.0053	7.2	0.15	98.0	8.5 ml/min 0.2M $NaHCO_3$ + 2 ml/l Triton X-100 + 5.0 g/l NaCl

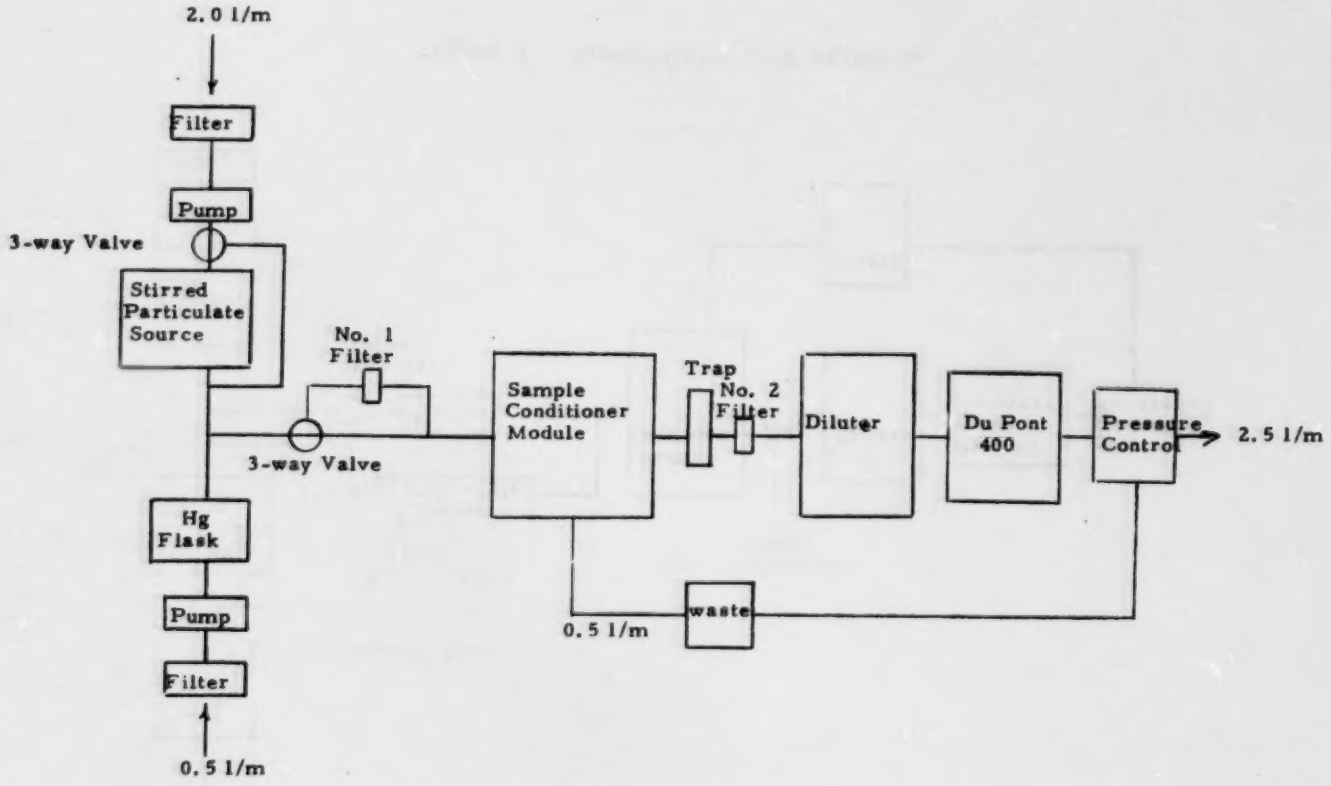


Figure 8. Particulate test assembly.

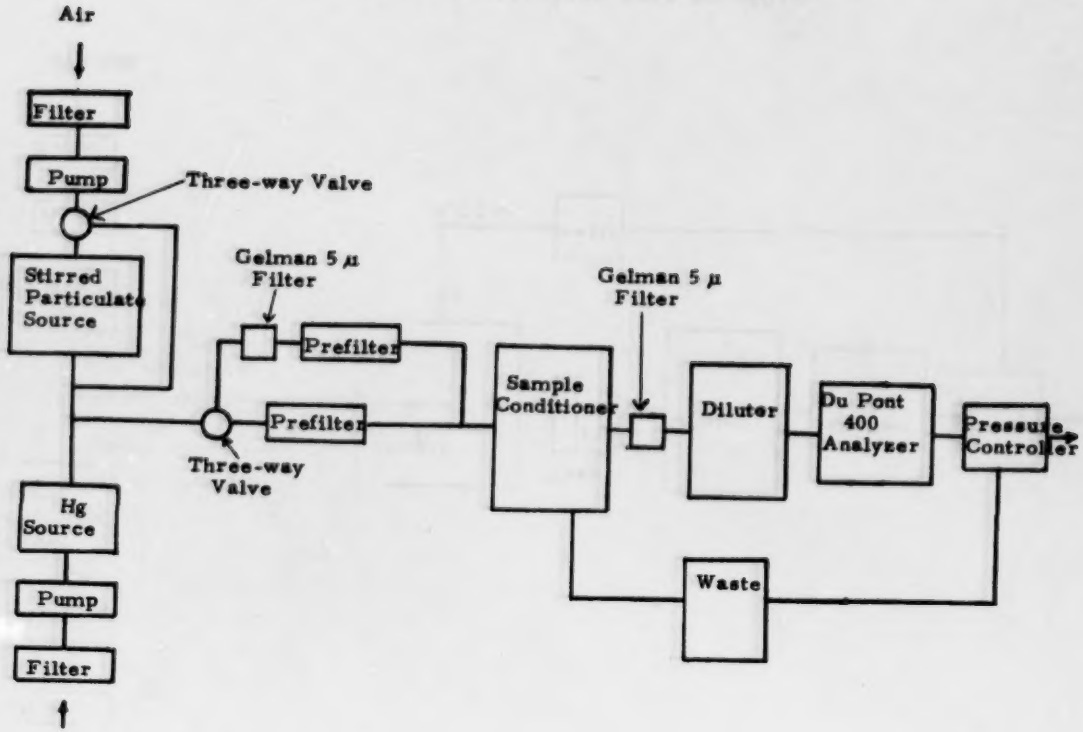


Figure 9. Particulate test assembly.

Tests 1-6 (Table 3) were conducted with the axis of the scrubber coil positioned vertically. The tests averaged 96.3% removal of the ash. In tests with the axis of the coil tilted ~20° from the vertical, removal efficiency increased to 97.8%. In the latter position, the gas and liquid passed as slugs through the coil. In tests 10 and 11 the detergent level caused the formation of a froth in the separator. Thus some of the liquid was entrained. The addition of salt together with a somewhat lowered level of Triton X-100 resulted in improved mechanical performance in the separator but the removal efficiency was not improved over Tests 7 and 8.

Since even the passage of ~2% of the fly-ash would prove a considerable operating problem during continuous operation, the effects of a filter between the conditioner and the diluter were examined.

Tests of the Conditioner-Prefilter Subsystem--

Introduction of a prefilter immediately prior to the sample conditioner simplifies the operation of the conditioner unit, which at high particle loading levels, tends to become clogged. A series of heated prefilter configurations were examined including: a ten inch section of cast iron pipe, an eight inch glass trap and a vertical stainless tube designed for easy removal of accumlated fly-ash. The latter design was selected. In use, it is hard plumbed to the conditioner unit and the major portion of collected fly-ash may be evacuated by removal of the plug attached at the bottom of the prefilter. It is sized so that at loadings in the range of 10-15 g/m^3 it must be emptied once in each 24 hours of operation.

The test assembly used in examining the effects of particulates during continuous mercury vapor analyses is shown schematically in Figure 8. In operation, the air containing mercury vapor and coal fly-ash from the stirred particles generator was passed directly through the prefilter at 2.5 liters/minute. The prefilter was maintained at 400°C by use of an external heating tape. For determinations of particle levels, a by-pass containing a Gelman 5 μ filter was added around the prefilter.

All tests were conducted to establish possible losses of mercury in the fly-ash. In general it was established that the prefilter temperature was critical in avoidance of mercury losses. At prefilter temperatures below 300°C some losses could be detected. At 400°C or higher, no measured losses were observed. The operational technique involved establishment of a constant mercury level through the system prior to introduction of fly-ash. This was compared with the results obtained when fly-ash was added to the system. Fly-ash levels were determined by use of Gelman (5 μ) 45 mm filters in front of the prefilter and behind the sample conditioner to establish the particulate removal efficiency. Deposits were quantitated by weight.

Data from a series of tests utilizing the system with prefilter are shown in Table 4. The prefilter and conditioner temperatures were established at 400°C in all tests. The scrubber used 5 ml/min of 0.2M $NaHCO_3$ to which 1 ml/liter of 10% Triton X-100 was added. In determinations of the quantities in the table: Mercury levels are estimated to have a coefficient of variation

TABLE 4. PARTICULATE REMOVAL DURING CONDITIONER/PREFILTER TESTS

Test No.	Mercury* Concentration ($\mu g/m^3$)	Particulate Input (g/m^3)	Particulate at Exit (g/m^3)	Particulate Removal Efficiency	System Pressure (cm H_2O)	Run Duration (min)
1 (a)	220	7.3	< 0.01	100%	800	25
(b)	220	0	0	--	800	--
2 (a)	90	6.5	< 0.01	100%	800	33
(b)	90	0	0	--	800	--
3 (a)	160	14.8	< 0.01	100%	800	25
(b)	160	0	0	--	800	--
4 (a)	45	10.6	< 0.01	100%	800	30
(b)	45	0	0	〃	800	--
5 (a)	770	9.6	< 0.01	100%	800	30
(b)	770	0	0	--	800	--
6 (a)	430	12.8	< 0.01	100%	800	30
(b)	430	0	0	--	800	--

* cv-2%

(CV) of 2%. Particulate concentrations have a CV of 6-10%. However, an absolute quantity of 0.0002g of fly-ash is detectible on the filter behind the sample conditioner. Thus, particle removal is essentially quantitative.

The data in Table 4 show highly satisfactory performance by the conditioner-prefilter system in removal of particulates from coal fly-ash at high loading levels.

Sulfur dioxide removal tests--The system with the prefilter was tested for SO_2 removal and for mercury passage using the assembly shown schematically in Figure 10. Initially, tests were run with the prefilter temperature at high levels (700-800°C); in this case, a non-condensible vapor resembling H_2SO_4 (probably SO_3) was generated. This vapor passed through the system to the DuPont 400 Photometer where it interfered with the determination of mercury. When the prefilter temperature was lowered to 400°C, the vapor disappeared as did the interference with the mercury analysis. At temperature < 300°C, some mercury appeared to be lost in the prefilter.

Quantitation of sulfur dioxide removal was achieved by use of sodium carbonate bubblers positioned on a by-pass around the prefilter and behind the conditioner. Sulfur dioxide levels in these tests were achieved by use of a tank source of SO_2 (Matheson) which was metered into the system by use of a needle valve and rotameter. These are not shown in Figure 9.

Sulfur dioxide concentrations from 220-2175 ppm were tested against mercury levels from 39 to 750 $\mu g/m^3$. Table 5 shows that sulfur dioxide removal was quantitative with the prefilter-conditioner system at all levels tested.

Removal of Particulates and Sulfur Dioxide by Prefilter-Conditioner--Using a combination of the two test systems previously described, tests with particulates and sulfur dioxide were carried out to determine whether mercury vapor could be transmitted without losses. The schematic of the test assembly is shown in Figure 11.

Input levels of fly-ash particulates and SO_2 were determined by use of a by-pass. Downstream levels behind the sample conditioner were similarly placed on a by-pass; no particles or SO_2 survived to this point in the test apparatus.

Tests were started by establishing and measuring each steady-state mercury vapor level with the DuPont Photometer. Fly-ash and SO_2 were added to the gas stream and Hg levels were redetermined. In Table 6, determination (b) for each test shows the steady level of mercury vapor before addition of particulates and SO_2. In no test did a variation outside the limits of the normal coefficient of variation of the mercury determination occur when particulates and SO_2 were added to the stream. In no case was SO_2 or fly-ash detected downstream from the conditioner.

The conditioner-prefilter configuration appeared to be most successful in these laboratory tests.

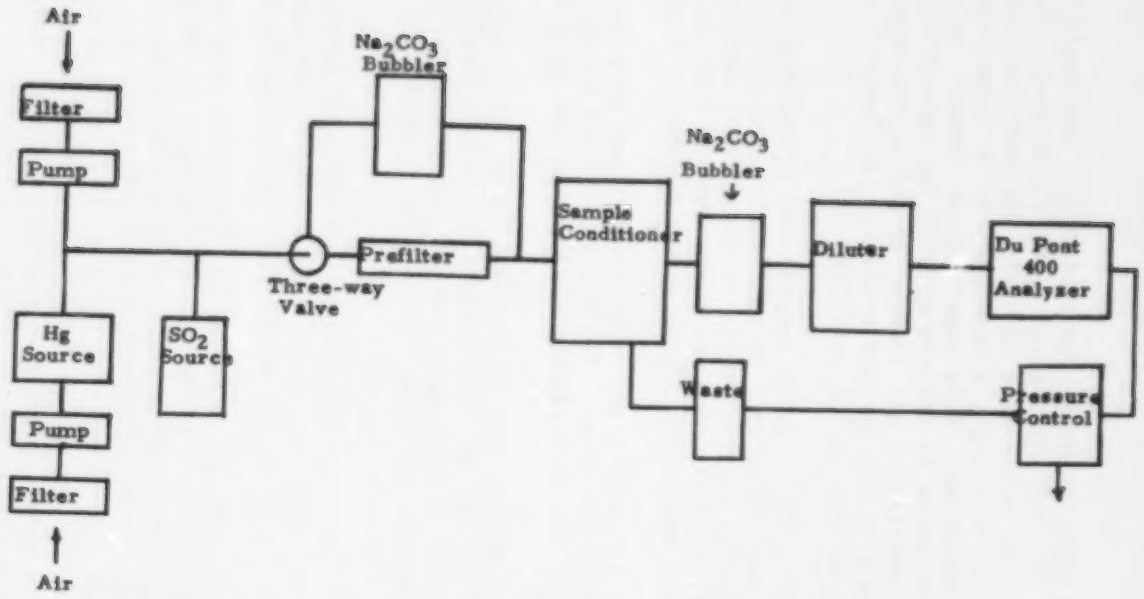


Figure 10. Sulfur Dioxide test assembly.

TABLE 5. SULFUR DIOXIDE REMOVAL DURING CONDITIONER TESTS

Test No.	Mercury Concentration ($\mu g/m^3$)*	SO_2 Conc. at Inlet (ppm)	SO_2 Conc. behind Conditioner (ppm)	Removal Efficiency** (%)	Duration of Test (min)	System Pressure (cm H_2O)
1	43	435	none detected	99.99	57.8	760
2	49	220	"	"	60	755
3	81	760	"	"	42	810
4	39	2175	"	"	25	810
5	41	750	"	"	46	810
6	205	410	"	"	50	810
7	225	720	"	"	44	810
8	195	2050	"	"	25	810
9	750	460	"	"	45	810
10	750	1960	"	"	30	810

* Estimated coefficient of variation 2%

** Detection limit is 10 μg of SO_4/20 ml of solution in bubbler

TABLE 6. REMOVAL OF SULFUR DIOXIDE AND PARTICULATES BY PREFILTER CONDITIONER

Test No.	Mercury Level* ($\mu g/m^3$)	Part. Conc. at Entry (g/m^3)	Part. Conc. behind Cond. (g/m^3)	SO_2 Conc. at Entry (ppm)	SO_2 Conc. behind Cond.** (ppm)	Part. Removal Efficiency(%)	SO_2 Removal Efficiency(%)	Duration of Test (min)	System Pressure (cm H_2O)
1 (a)	81	7.6	0	110	0	100	100	22	800
(b)	81	--	--	--	--	--	--	--	"
2 (a)	91	12.7	0	210	0	100	100	30	800
(b)	91	--	--	--	--	--	--	--	"
3 (a)	83	12.2	0	1040	0	100	100	25	800
(b)	83	--	--	--	--	--	--	--	"
4 (a)	79	11.6	0	410	0	100	100	24	800
(b)	79	--	--	--	--	--	--	--	"
5 (a)	443	12.9	0	420	0	100	100	25	800
(b)	443	--	--	--	--	--	--	--	"
6 (a)	790	9.8	0	480	0	100	100	25	800
(b)	790	--	--	--	--	--	--	--	"
7 (a)	950	12.3	0	230	0	100	100	25	800
(b)	950	--	--	--	--	--	--	--	"

* Coefficient of variation estimate 2%.

** SO_2 is undetectable; removal <99.99%.

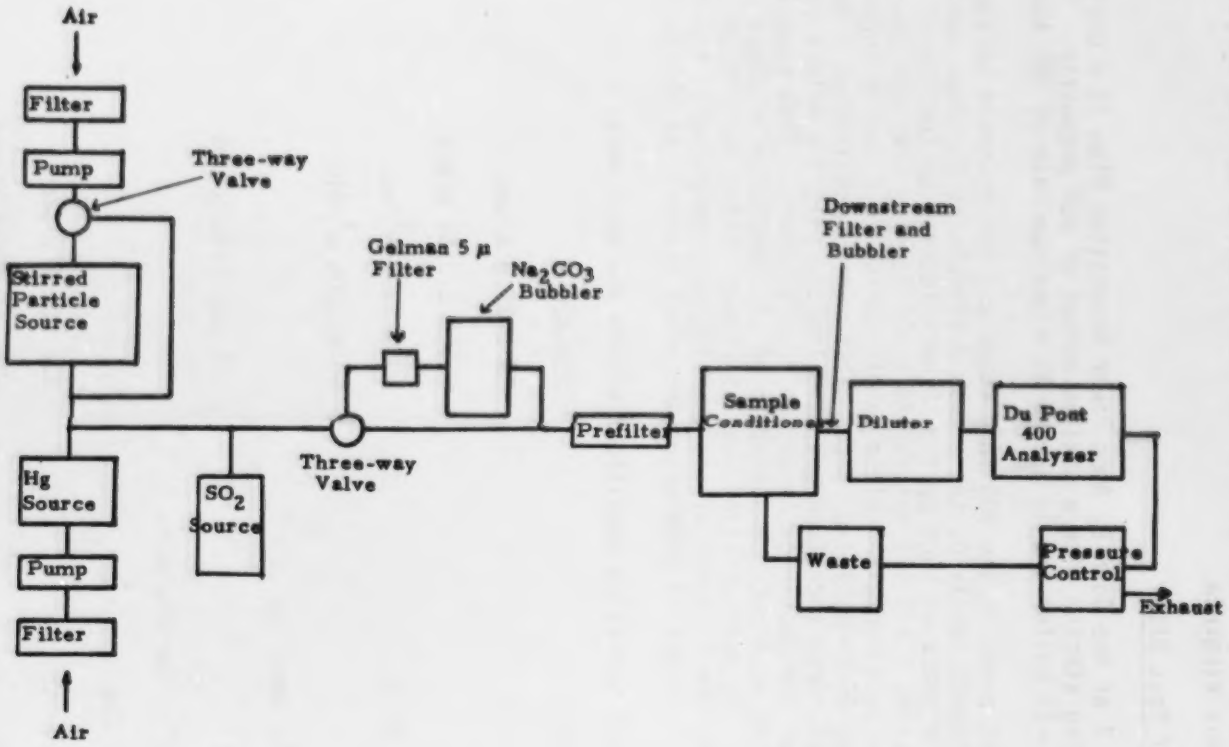


Figure 11. Combined Sulfur Dioxide and Particulate test assembly.

FIELD TESTS

During April, 1976 the interface system was tested in conjunction with determinations of mercury in the exhaust gases of the No. 2 Unit of the J.E. Amos Power Generating Plant of the American Electric Service Corporation at St. Albans, West Virginia.

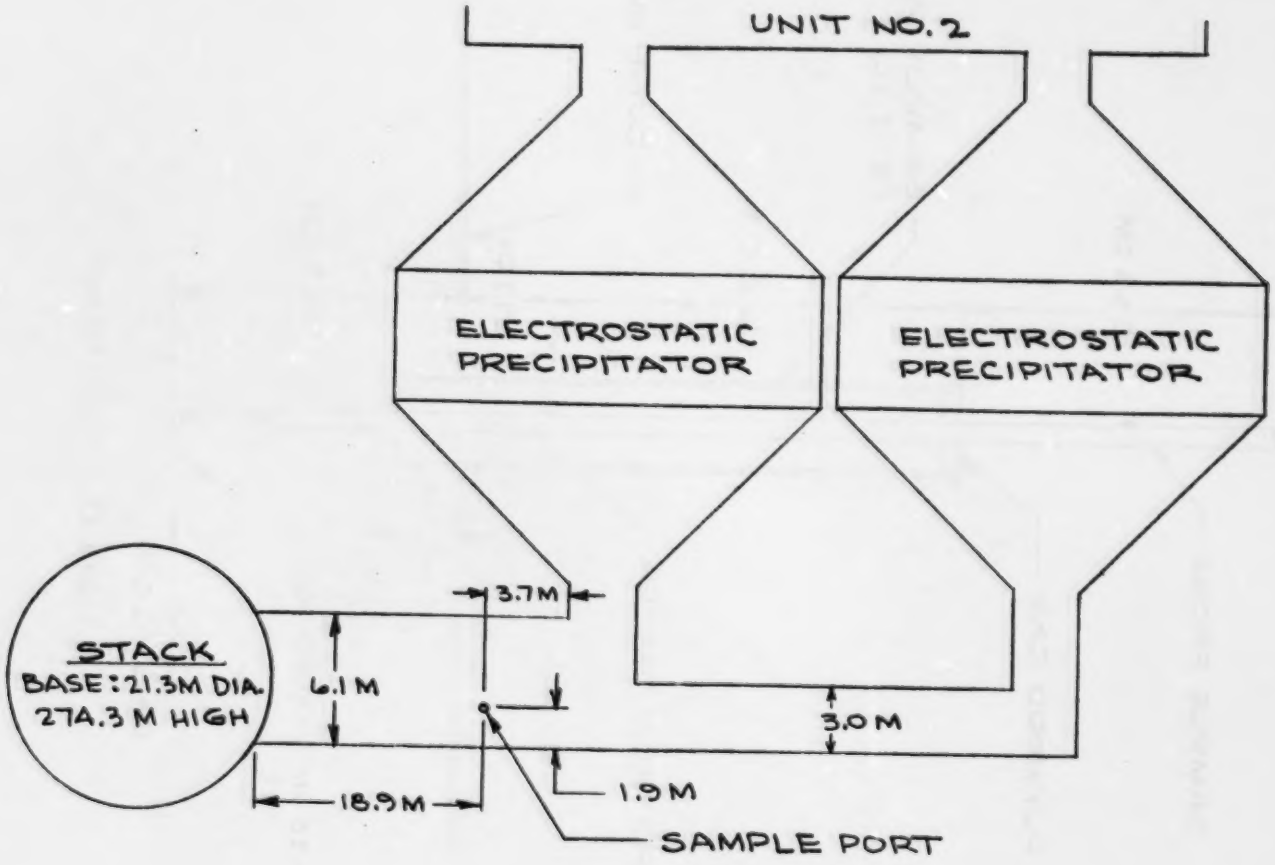
Description of Test Site

Unit No. 2 of the John E. Amos Power Generation Plan is a coal-fired steam generating station with a maximum output of 800 megawatts. The coal used contains ~1% sulfur and is burned at a maximum rate of 350 tons per hour.

Testing at ports in the 900 foot stack was not possible because of the amount of equipment involved, the access difficulties to stack ports and the limited catwalk space at such ports. A port located on the top of a horizontal duct measuring 6.1 x 6.1 meters (20 x 20) feeding into the stack was selected. The duct contained eight sample ports, 76.2 cm (30 inches) high made of 12.7 cm (5 inches) i.d. pipe. These were installed at a point 18.9 meters (62 feet) from the stack but were located only 3.6 meters (12 feet) from the junction of the duct from those two precipitators. Flow anomalies caused by this location were not considered to be of importance in these mercury vapor measurements. The port utilized was 1.9 meters (6-1/4 feet) from the duct wall. Figure 12 shows the location of the sampling port relative to the stack and precipitators. Figure 13 dimensions the probe assembly at the port.

The general operating conditions within the duct were:

Temperature:	280-320 F
Air Velocity:	26.8-35.8 m/sec
or:	1,610-2, 150 m/min
Gas Flow Rate:	1000-1300 m^3/sec
or:	60-80,000 m^3/min
Isokinetic Sampling	
Rate:	114-150 liters/min
(with 3/8" probe tip diam.)	
Estimated Gas	
Components:	NO_x: to 600 ppm
	SO_2: to 400-600 ppm



SCHEMATIC DIAGRAM OF SAMPLING PORT LOCATION

Figure 12. John E. Amos Plant, Unit #2, 800 Megawatt output.

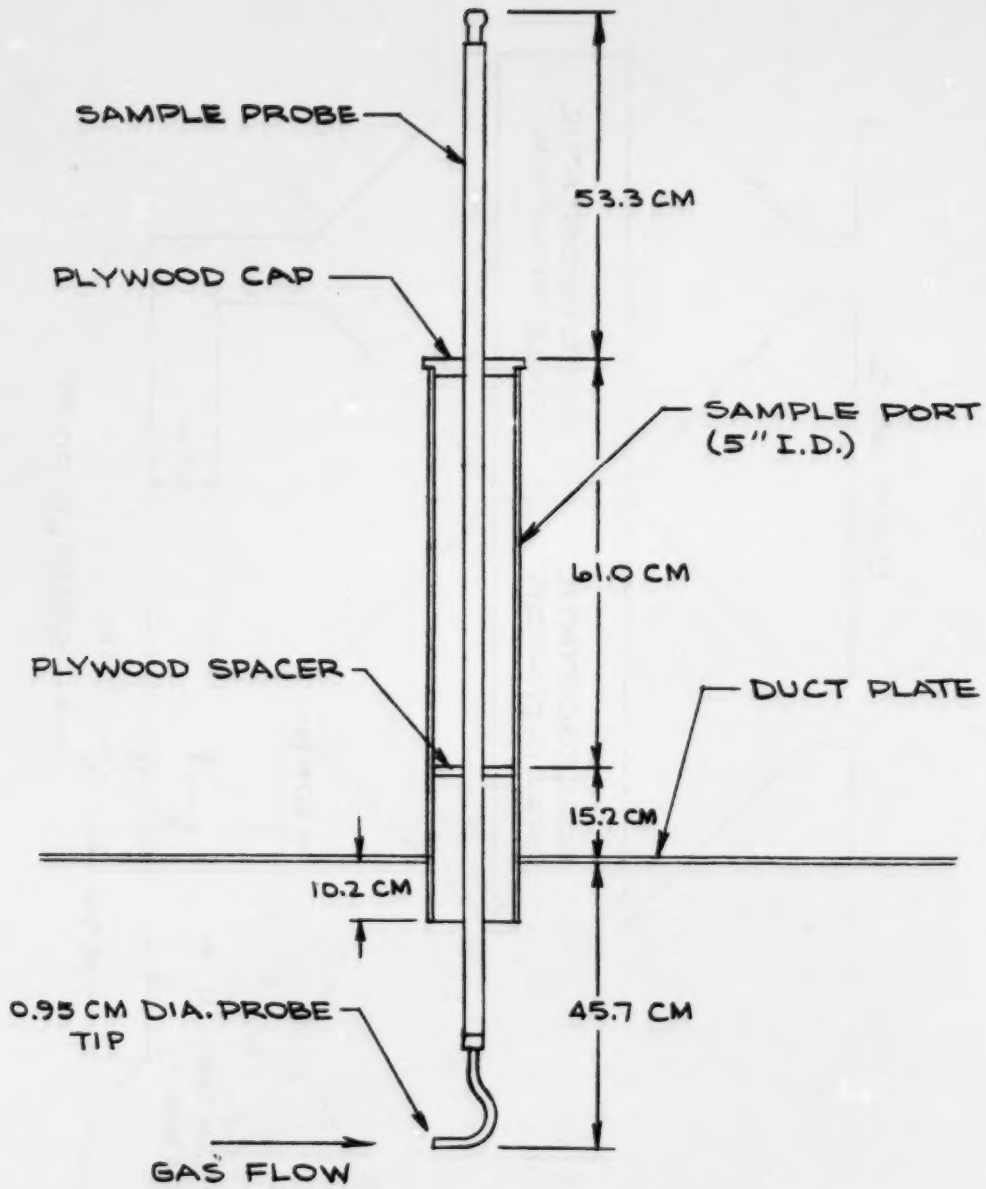


Figure 13. Probe alignment.

Estimated Particulate

Level: 0.5-1.0 g/m^3

Test Assembly

To obtain representative samples, a six foot, heated, glass lined, stainless steel probe was used. The probe extended 41.9 cm (16.5 inches) into the duct and was fitted to the sample port by means of plywood spacers. The probe was heated using a 10 amp (120 V) variac set at 40-50 divisions which maintained a temperature of approximately 300 F at the probe wall. The duct gas temperature was 280-320 F.

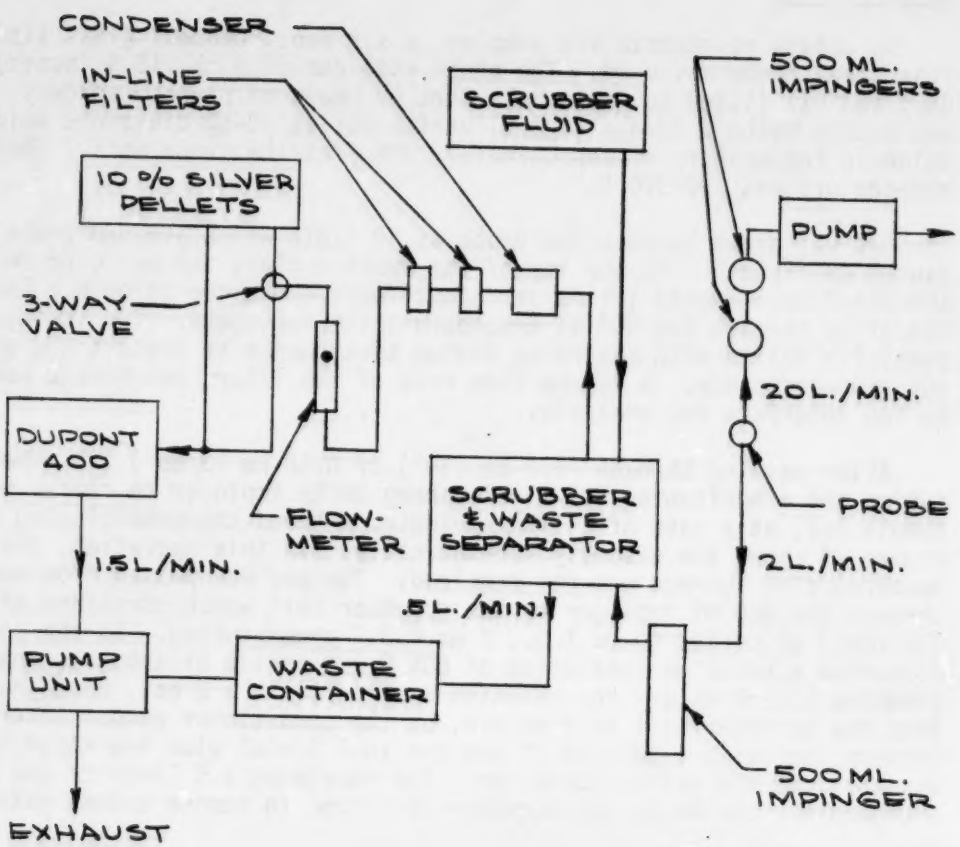
Gas was drawn through the probe at 22 l/min which avoided probe clogging caused by fly-ash. At the top of the probe a glass tee was used to divide the air flow. Twenty liters per minute were exhausted through a Thomas pump operating through two 500 ml Greenberg-Smith impingers. The impingers were partially filled with saturated sodium bicarbonate to protect the pump from SO_2 and condensate. A volume flow rate of two liters per minute was diverted to the interface for analysis.

After passing through 76.2 cm (30") of 0.48 cm (3/16") I.D. amber latex tubing and a horizontal 500 ml Greenberg-Smith impinger to remove water, the sample gas, at a rate of 2 liters/minute, entered the conditioning unit. Figure 14 shows the assembly schematically. In this operation, the thermal decomposition furnace was not required. The gas was passed from the probe through the 500 ml impinger to the scrubber coil which consisted of 3.66 m (12 feet) of coiled (5 mm I.D., 7 mm O.D.) pyrex tubing. In the coil the gas contacted a solution consisting of 80% 0.2 M sodium bicarbonate and 20% 0.2 M ammonium bicarbonate. The solution was stored in a 2 gal. container and pumped into the scrubber coil at 5 ml/min. by the conditioner pump. After passing through the coil, a portion of the gas (0.5 l/min) plus the waste liquid was drawn off to a 2 gallon container. The remaining 1.5 l/min of gas went through a condenser, cooled by the scrubber solution, to remove excess water in the gas.

Two heated 25 mm Gelman in-line filter holders containing Whatman #41 paper removed any residual fly-ash before the rotameter. The gas was directed by means of a three way valve to the DuPont 400 spectrophotometer or through 10% silver alumina tablets and thence to the photometer. The DuPont 400 used directly gave the measurement of the mercury yielding a base line or zero for comparison when mercury-free gas was needed for passage through the photometer. The difference between the two photometer readings was calibrated by putting a known amount of mercury through the system.

The gas was exhausted through the pump unit. This unit served to keep the pressure constant in the photometer cell by means of a pressure regulator. Also, it pulled 0.5 l/min. of gas through the waste container.

To check the results against the reference method, measured volumes of gas were drawn through midget impingers connected downstream of the probe. After removing the SO_2 in the gas by passing it through saturated sodium bicarbonate



NOTES:

Scrubber Fluid - 80% 0.2M $NaHCO_3$ & 20% (0.2M NH_4CO_3)

Pumping Rate: 4.8 ml/min

Lines from probe to scrubber are amber latex rubber tubing

Gelman In-Line Filter Holders contained Whatman #41 25 mm Filter Paper

Probe Heater - Variac setting 40

Figure 14. Detailed schematic of measurement system.

in the first midget impinger, mercury was collected in a nitric acid-potassium permangate solution. The amount of mercury was determined by using the photometer section of a GEOMET Model 103 Mercury Monitor to measure the peak obtained from a modified Hatch and Ott procedure.

Operation of System

Maintenance--

Each morning the lines and impinger between the probe and scrubber were rinsed with distilled water. Fly-ash was removed from the top of the probe, and the 20 liter per minute exhaust line and impingers were cleaned. Every two hours new filter paper was placed in the in-line filters. The first filter after the condenser restricted the air flow (because of collected fly-ash) if left for long periods. Periodic air flow checks at the probe were also necessary since the Thomas pump on the exhaust may cause the rotameter in the measurement side of the system to give erroneous readings when variations from the inital pressure drop ratio take place.

Test Operations--

In the tests conducted at the duct of the J.E. Amos Power Generation Plant the results obtained by use of the instrumentation comprising the interface system and the DuPont 400 Photometer Analyzer were compared with determinations obtained by use of a simple absorption train together with conven tional reduction and measurement procedures. Details of the procedures have been included in Appendix B.

The mercury level as determined by the instrumentation was zeroed against the signal obtained by diverting the air flow through 10% silver-alumina tablets. The signal difference as measured by the DuPont Photometer was calibrated by introduction of known quantities of mercury vapor into the system at the probe. This internal calibration technique was also utilized to check the reproducibility of the operation from one test to another.

The stack mercury concentration was determined separately by hooking a permanganate bubbler (1 ml 0.25 M $KMnO_4$, 2.5 ml 1:1 HNO_3 diluted to 15 ml) to a saturated $NaHCO_3$ bubbler and connecting them directly to the probe. Sampling intervals of 2.5 minutes were found to be about the best length of time for these independent sample collections.

For comparison, a permanganate/acid bubbler was also used to collect various volumes of mercury vapor introduced into the line behind the probe. This was used to quantitate calibration peaks.

To analyse the bubblers, the contents were reduced (3 ml 20% Wt./Vol. $SnCl_2$ in 6N HCl) and caught in a second permanganate bubbler (0.3 ml 0.25 M $KMnO_4$, 2 ml 1:1 HNO_3 diluted to 15 ml). The second bubbler was placed in series with an $NaBH_4$ bubbler (~1 g/15 ml), and empty bubbler and the Geomet analyzer, on reduction with 2 ml of $SnCl_2$ the vapors were tranferred utilizing an air flow of 2 l/min to the Geomet Model 103. The amount of mercury was determined using peak heights from prepared calibration curves.

Results

The results obtained in thirty-seven comparisons between measurements made with the DuPont Photometric Analyzer and a direct bubbler measurement of mercury in the stack (duct) are tabulated in Table 7. The three right hand columns summarize the comparisons. Table 8 is a summary of the calibration test data (indicated by the designation P) and bubbler tests on the stack gases (designated by S). All Run # designations correspond with the samples on Table 7. While samples were not collected simultaneously with test runs they were collected immediately before or after the determination by the photometer.

Table 9 defines the terms used in Tables 7 and 8.

Discussion of Results

The results obtained and listed in Table 7 show that a good calibration between the data obtained with the automated system and the bubbler technique has been obtained. In the majority of the test runs, the two methods agreed very well with respect to the amount of mercury in the stack gas. In a few cases a significant difference appeared.

Photometric measurements with the interface and the DuPont Analyzer were made considerably more easily than those with the bubbler. By this technique, continuous monitoring of the effluent stream was clearly possible.

In general, the concentrations of mercury vapor in the exhaust gases were relatively low, ranging from 1.74 to 6.96 ug/m^3 in determinations made by the instrumented system. The range was 1.60 to 7.25 in measurements made by the bubbler techniques. The mean levels were 4.23 $\pm$ 1.28 ug/m^3 (instrumental) and 4.66 $\pm$ 1.42 ug/m^3 (bubbler). The mean values represent the entire set of data collected from 4/21 through 4/30.

These tests were conducted in the presence of relatively high levels of potential interferences: 0.5 - 1.0 g/m^3 of particulates, ~400-500 ppm SO_2 and ~600 ppm NO_x. The levels of SO_3 were not defined. The presence of SO_3 necessitated the changes in the experimental assembly involving the silver-alumina tablets and the zero baseline determination. However, after incorporating these modifications, the field performance of the interface-photometer system was approximately equal to that of the laboratory-tested configuration discussed in above. Response and fall times remained within the ranges previously measured. The detection limit was etimated at 0.4-0.5 ug/m^3, as previously. Detailed replication of the field tests were not conducted; no reason for a changed precision estimate was apparent. A coefficient of variation in the 5-6% range seems predictable.

The performance stability and reliability of the system were exceptional, particularly when compared with manual methods.

Accuracy as determined by comparison of results obtained with the instrumentation and with the manual method appears more than adequate. However,

TABLE 7. TESTS OF AUTOMATED INTERFACE INSTRUMENTATION

Run	Date	Time	Peak Area in Squares	Calc. Stack Hg Peak Height in Squares	DuPont Flow Rate l/min	Content Bubbler in ng	of Peak Calib. in ng/l	Calibration Factor in $ng^{-1}-l^{-1}-sq^{-1}$	Stack Hg Signal Height in Squares	Bubbler Hg Conc of stack in $\mu g/m^3$	Photometric Hg Conc of stack calculated in $\mu g/m^3$	Ratio Bubbler to Photometric Conc. of Hg in stack
1			719	35.95	1.5	52.5	94.5	2.63	1	1.90	2.63	0.722
2			857.5	42.88	1.5	34.0	61.2	1.43	--	--	--	--
3	4/21	15:35	2907.5	72.69	1.5	42.5	38.25	0.526	3.3	1.60	1.74	0.920
4	4/22	10:25	1381	27.62	1.3	16.5	17.13	0.620	0.0	0.00	0.00	0.000
5	4/22	15:03	2802.5	62.28	1.3	39.5	36.46	0.585	3.0	1.97	1.76	1.12
6	4/22	17:23	2277.5	56.94	1.3	36.5	37.90	0.666	3.3	2.50	2.20	1.14
7	4/23	10:30	1705	37.89	1.3	28.5	26.31	0.694	6.5	5.29	4.51	1.17
8	4/23	17:10	2222.5	55.56	1.5	48.0	43.20	0.778	4.6	4.42	3.58	1.23
9	4/24	10:15	2017.5	40.35	1.5	34.5	24.84	0.616	7.3	4.17	4.50	0.927
10	4/24	14:45	2482.5	62.06	1.7	39.0	30.97	0.499	7.0	3.01	3.49	0.862
11	4/24	16:47	2712.5	67.81	1.5	36.0	32.40	0.478	4.0	2.65	1.91	1.39
12	4/26	15:05	928	23.2	1.25	14.0	14.54	0.627	7.0	3.99	4.39	0.909
13	4/26	16:06	869	21.73	1.5	12.0	10.80	0.497	7.0	4.22	3.48	1.21
14	4/27	10:37	766	19.15	1.5	14.0	12.60	0.658	5.0	3.35	3.29	1.02
15	4/27	12:39	1429	35.73	1.5	11.0	9.90	0.277	10.0	6.50	2.77	2.35
16	4/27	15:19	1247	31.18	1.5	12.5	11.25	0.361	10.0	5.55	3.61	1.54
17	4/27	16:45	1402	35.05	1.5	13.5	12.15	0.347	8.5	5.40	2.95	1.83
18	4/27	17:50	1241	31.03	1.5	14.0	12.60	0.406	9.6	5.00	3.90	1.28

(continued)

TABLE 7 (continued)

Run	Date	Time	Peak Area in Squares	Calc. Stack Hg Peak Height in Squares	DuPont Flow Rate l/min	Content Bubbler in ng	of Peak Calib. in ng/l	Calibration Factor in ng^{-1}-sq^{-1}	Stack Hg Signal Height in Squares	Bubbler Hg Conc of stack in $\mu g/m^3$	Photometric Hg Conc. of stack calculated in $\mu g/m^3$	Ratio Bubbler to Photometric Conc. of Hg in stack
19	4/28	08:00	754	18.85	1.5	11.5	10.35	0.549	9.3	5.00	5.11	0.978
20	4/28	09:08	760	19.00	1.5	11.5	10.35	0.545	8.5	4.75	4.63	1.03
21	4/28	10:00	1710	42.75	1.5	21.5	19.35	0.453	11.3	4.75	5.12	0.928
22	4/28	11:24	1033	25.83	1.5	11.5	10.35	0.401	12.5	4.50	5.01	0.898
23	4/28	14:25	866	21.65	1.5	12.0	10.80	0.499	9.3	4.95	4.64	1.07
24	4/28	15:09	791	19.78	1.5	11.0	9.90	0.501	8.5	4.20	4.26	0.986
25	4/28	17:53	1127	28.18	1.5	15.5	13.95	0.495	8.6	3.80	4.26	0.892
26	4/29	09:50	895	22.38	1.5	13.0	11.70	0.523	13.3	7.25	6.96	1.04
27	4/29	11:30	1262	31.55	1.5	19.75	17.78	0.564	11.6	6.50	6.54	0.994
28	4/29	13:03	1654	41.35	1.5	20.5	18.45	0.446	11.3	5.05	5.04	1.002
29	4/29	15:10	1095	27.38	1.5	25.0	22.50	0.822	5.75	5.61	4.73	1.19
30	4/29	17:20	2140	53.50	1.5	30.5	27.45	0.513	8.3	4.85	4.26	1.14
31	4/29	18:16	2167.5	61.93	1.5	29.0	26.10	0.421	11.5	6.75	4.84	1.39
32	4/30	08:30	1522	33.82	1.5	20.0	16.00	0.473	10.66	5.05	5.04	1.002
33	4/30	10:05	1098	24.40	1.5	14.5	11.60	0.475	11.0	5.95	5.23	1.14
34	4/30	12:50	1895	47.38	1.5	28.75	25.88	0.546	9.5	5.30	5.19	1.02
35	4/30	14:35	1540	44.00	1.5	33.50	34.47	0.783	8.0	6.90	6.26	1.10
36	4/30	16:03	1870	53.43	1.5	38.50	39.62	0.742	6.8	5.50	5.05	1.09
37	4/30	17:03	2562.5	73.21	1.5	43.50	44.76	0.611	8.5	5.15	5.19	0.992

TABLE 8. CALIBRATION AND DIRECT BUBBLER MEASUREMENTS

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	DuPont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
1-A		P	135		1.5	52.5		
1-B		S	135	2		52.5		2.5
1-C		S	30	2		9.5	1.90	2.5
2-A		S	25	2		7.5	1.50	2.5
2-B		P	90		1.5	34.0		
3-A	4/21	P	111		1.5	42.5		
3-B	4/21	S	27	2		8.0	1.60	2.5
4-A	4/22	S	0	1.6		0.0	0.00	2.5
4-B	4/22	P	47		1.3	16.5		
5-A	4/22	P	103		1.3	39.5		
5-B	4/22	S	24	1.6		7.0	1.75	2.5
5-C	4/22	S	32	1.6		10.5	2.18	3.0
6-A	4/22	P	96		1.3	36.5		
6-B	4/22	S	32	1.6		10.5	2.62	2.5
6-C	4/22	S	30	1.6		9.5	2.38	2.5
7-A	4/23	P	76		1.3	28.5		
7-B	4/23	S	58	1.6		21.0	5.29	2.5
8-A	4/23	P	124		1.5	48.0		
8-B	4/23	S	58	1.9		21.0	4.42	2.5

*P= Calibration tests by injection of vapor which passed thru as peak. S= Bubbler Stack Samples

(continued)

TABLE 8 (continued)

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	DuPont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
9-A	4/24	P	91		1.5	34.5		
9-B	4/24	S	57	1.9		20.5	4.33	2.5
9-C	4/24	S	56	2.0		20.0	4.00	2.5
10-A	4/24	P	102		1.7	39.0		
10-B	4/24	S	45	2.2		15.5	2.82	2.5
10-C	4/24	S	50	2.2		17.5	3.19	2.5
11-A	4/24	P	95		1.5	36.0		
11-B	4/24	S	37	1.8		12.0	2.67	2.5
11-C	4/24	S	38	1.9		12.5	2.63	2.5
12-A	4/26	S	42	1.7		14.5	3.41	2.5
12-B	4/26	S	57	1.5		20.5	4.56	3.0
12-C	4/26	P	41		1.3	14.0		
13-A	4/26	S	53	1.8		19.0	4.22	2.5
13-B	4/26	S	78	1.8		29.0	6.44	2.5
13-C	4/26	P	36		1.5	12.0		
14-A	4/27	P	41		1.5	14.0		
14-B	4/27	S	71	2.0		16.5	3.30	2.5
14-C	4/27	S	73	2.0		17.0	3.40	2.5
15-A	4/27	P	34		1.5	11.0		

*P = Calibration tests by injection of vapor which passed thru as peak. S= Bubbler Stack Samples

(continued)

TABLE 8 (continued)

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	DuPont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
15-B	4/27	S	88	2.0		33.5	6.70	2.5
15-C	4/27	S	83	2.0		31.5	6.30	2.5
16-A	4/27	P	37		1.5	12.5		
16-B	4/27	S	76	2.0		28.5	5.70	2.5
16-C	4/27	S	72	2.0		27.0	5.40	2.5
17-A	4/27	P	40		1.5	13.5		
17-B	4/27	S	74	2.0		27.5	5.50	2.5
17-C	4/27	S	71	2.0		26.5	5.30	2.5
18-A	4/27	P	41		1.5	14.0		
18-B	4/27	S	69	2.0		25.5	5.10	2.5
18-C	4/27	S	67	2.0		24.5	4.90	2.5
19-A	4/28	P	35		1.5	11.5		
19-B	4/28	S	68	2.0		25.0	5.00	2.5
19-C	4/28	S	68	2.0		25.0	5.00	2.5
20-A	4/28	P	35		1.5	11.5		
20-B	4/28	S	53	2.0		19.0	3.80	2.5
20-C	4/28	S	76	2.0		28.5	5.70	2.5
21-A	4/28	P	59		1.5	21.5		
21-B	4/28	S	62	2.0		22.5	4.50	2.5

*P= Calibration tests by injection of vapor which passed thru as peak. S=Bubbler Stack Samples

(continued)

TABLE 8 (continued)

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	DuPont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
21-C	4/28	S	68	2.0		25.0	5.00	2.5
22-A	4/28	P	35		1.5	11.5		
22-B	4/28	S	54	2.0		19.5	3.90	2.5
22-C	4/28	S	69	2.0		25.5	5.10	2.5
23-A	4/28	P	36		1.5	12.0		
23-B	4/28	S	65	2.0		24.0	4.80	2.5
23-C	4/28	S	69	2.0		25.5	5.10	2.5
24-A	4/28	P	34		1.5	11.0		
24-B	4/28	S	59	2.0		21.5	4.30	2.5
24-C	4/28	S	57	2.0		20.5	4.10	2.5
25-A	4/28	P	45		1.5	15.5		
25-B	4/28	S	53	2.0		19.0	3.80	2.5
25-C	4/28	S	53	2.0		19.0	3.80	2.5
26-A	4/29	P	39		1.5	13.0		
26-B	4/29	S	96	2.0		36.5	7.30	2.5
26-C	4/29	S	95	2.0		36.0	7.20	2.5
27-A	4/29	P	55		1.5	19.75		
27-B	4/29	S	83	2.0		31.5	6.30	2.5
27-C	4/29	S	89	2.0		33.5	6.70	2.5

*P= Calibration tests by injection of vapor which passed thru as peak. S=Bubbler Stack Samples

(continued)

TABLE 8 (continued)

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	DuPont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
28-A	4/29	P	57		1.5	20.5		
28-B	4/29	S	64	2.0		23.5	4.70	2.5
28-C	4/29	S	73	2.0		27.0	5.40	2.5
29-A	4/29	P	68		1.5	25.0		
29-B	4/29	S	44	1.6		15.0	3.75	2.5
29-C	4/29	S	93	1.9		35.5	7.47	2.5
30-A	4/29	P	81		1.5	30.5		
30-B	4/29	S	64	2.0		23.5	4.70	2.5
30-C	4/29	S	68	2.0		25.0	5.00	2.5
31-A	4/29	P	78		1.5	29.0		
31-B	4/29	S	89	2.0		33.5	6.70	2.5
31-C	4/29	S	90	2.0		34.0	6.80	2.5
32-A	4/30	P	56		1.5	20.0		
32-B	4/30	S	66	2.0		24.0	4.80	2.5
32-C	4/30	S	71	2.0		26.5	5.30	2.5
33-A	4/30	P	42		1.5	14.5		
33-B	4/30	S	81	2.0		30.5	6.10	2.5
33-C	4/30	S	78	2.0		29.0	5.80	2.5
34-A	4/30	P	77		1.5	28.75		

*P= Calibration tests by injection of vapor which passed thru as peak. S- Bubbler Stack Samples

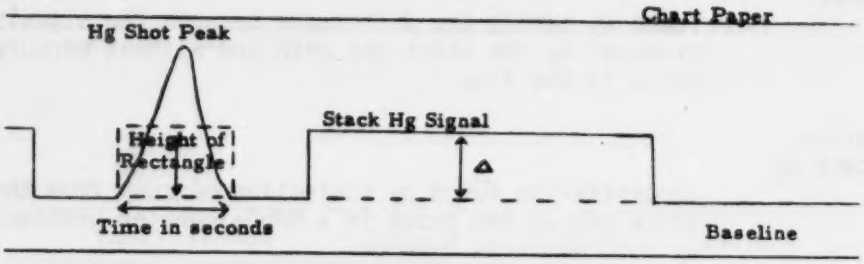
(continued)

TABLE 8 (continued)

Run No.	Date	Type*	Bubbler DVM Reading	Sample Flow Rate l/min	Du Pont Flow Rate l/min	ng Hg	Concentration Hg ng/l	Time minutes
34-B	4/30	S	62	2.0		22.5	4.50	2.5
34-C	4/30	S	81	2.0		30.5	6.10	2.5
35-A	4/30	P	88		1.5	33.5		
35-B	4/30	S	91	2.0		34.5	6.90	2.5
35-C	4/30	S	191	2.0		75.5	15.10	2.5
36-A	4/30	P	101		1.5	38.5		
36-B	4/30	S	75	2.0		28.0	5.60	2.5
36-C	4/30	S	72	2.0		27.0	5.40	2.5
37-A	4/30	P	113		1.5	43.5		
37-B	4/30	S	68	2.0		25.0	5.00	2.5
37-C	4/30	S	71	2.0		26.5	5.30	2.5

*P= Calibration tests by injection of vapor which passed thru as peak. S=Bubbler Stack Samples

TABLE 9. DEFINITION OF TERMS.



Peak Area-	Number of squares under the peak resulting from a mercury shot.
Stack Hg Peak-	Calculated by dividing PEAK AREA by time in seconds where time corresponds to the base of the peak and the length of a rectangle possessing the same area as the peak. The quotient is the width of the rectangle. This width would be the same as the height of a stack mercury signal resulting from the same level of mercury which produced the peak. Width = Area ÷ length
DVM Reading-	Reading on the GEOMET 103 produced by a 1 cc shot of Hg vapor collected in a $KMnO_4$ bubbler. This corresponds to the mercury level used to give the peak.
Hg Content of Bubbler-	Amount of mercury in ng corresponding to the DVM reading on the DVM - ng Hg calibration curve.
Hg Concentration of Peak-	Concentration of mercury which produced the Hg SHOT PEAK. Found by dividing Hg CONTENT OF BUBBLER by the time in seconds which corresponds to the base of the peak then multiplying by the inverse of the gas flow rate and a correction factor, 0.9, for the error in the chart speed. $[(ng\ Hg/sec)\ (1/sec)^{-1}\ 0.9]$

(continued)

Table 9-Continued

DEFINITION OF TERMS

Calibration Factor-	Calculated by dividing Hg CONCENTRATION OF THE PEAK by STACK Hg PEAK.
Stack Hg Signal Height-	Found by taking the difference between the signals/ produced by the stack gas with and without mercury pills in the line.
Hg Concentration of the stack by Bubbler-	Concentration found by collecting mercury from the stack gas at the probe in a $KMnO_4$ bubbler preceeded by a bicarbonate bubbler.
Hg Concentration of Stack Calculated-	Found by multiplying STACK Hg SIGNAL HEIGHT by the CALIBRATOR FACTOR.

determinations made by the bubbler-manual precedures may not be appropriate criteria for such comparisons.

In summary, the interface instrumentation appears to perform as required. It will permit the continuous monitoring of stack gases for mercury. The flexible nature of its design will permit its use in a wide variety of applications.

Appendix A

OPERATING INSTRUCTIONS

SECTION A1

CONTROLS AND CONNECTIONS

This appendix gives in detail the use of controls and connections to be made in operating the Interface System. A detailed description will be given of the Conditioning Module, the Diluter Module and the Pump Module.

The Conditioner Module

There are five operating controls on the conditioning module. Four of these on the front panel are shown in Figure A-1. They are: the power switch, the heater switch, the fluid switch, and the pyrometer set point knob. The fifth is the fluid pump stroke adjustment knob which is located inside the unit.

In addition there are 4 line connections on the unit. These are shown in Figure A-2.

The Power Switch--
Power to the unit is controlled by the red toggle switch on the upper right hand side of the front panel. In the "up" position, power is supplied to the solid state electronic power supply. This permits the other switches to function, and it activates the Rotron cooling fan in the side of the unit.

The light above the switch indicates that the power supply has been activated.

The Heater Switch--
Power is supplied to the unit by connecting the 3 wire electrical cord to the A.C. Connector in the lower left hand corner of the panel. 60 Hz, 110 VAC power is required. Power to the furnace heater is controlled by the red toggle switch which is labelled "Heater". In the "up" position the power is supplied to the heater.

The light above the switch indicates that power is being supplied to the heater.

The Fluid Switch--
Power to the fluid pump is controlled by the white toggle switch. In the "up" position the pump is activated and pumps fluid into the scrubber coil. The light above the switch indicates that the pump is on.

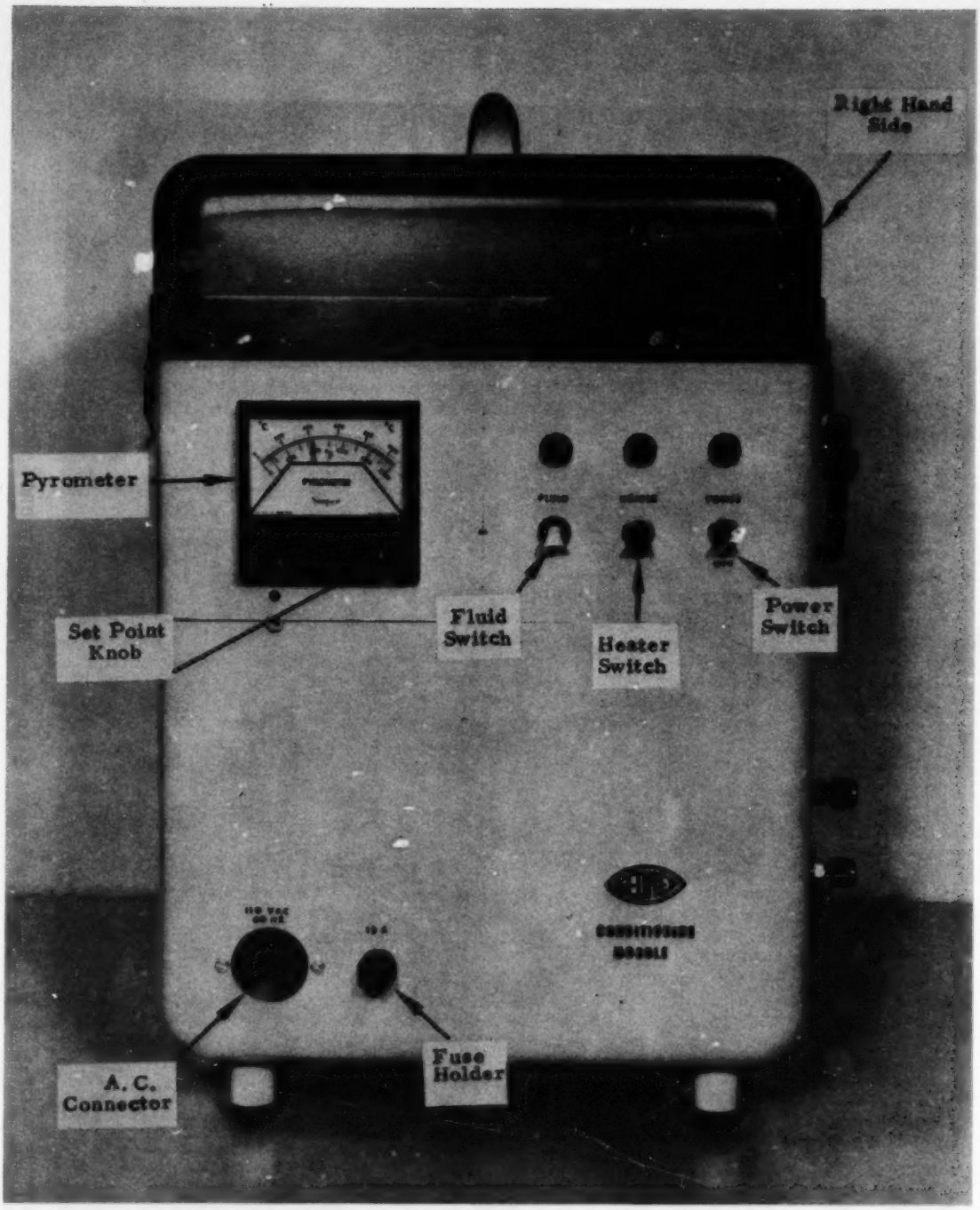


Figure A-1. Front Face of Conditioner Module

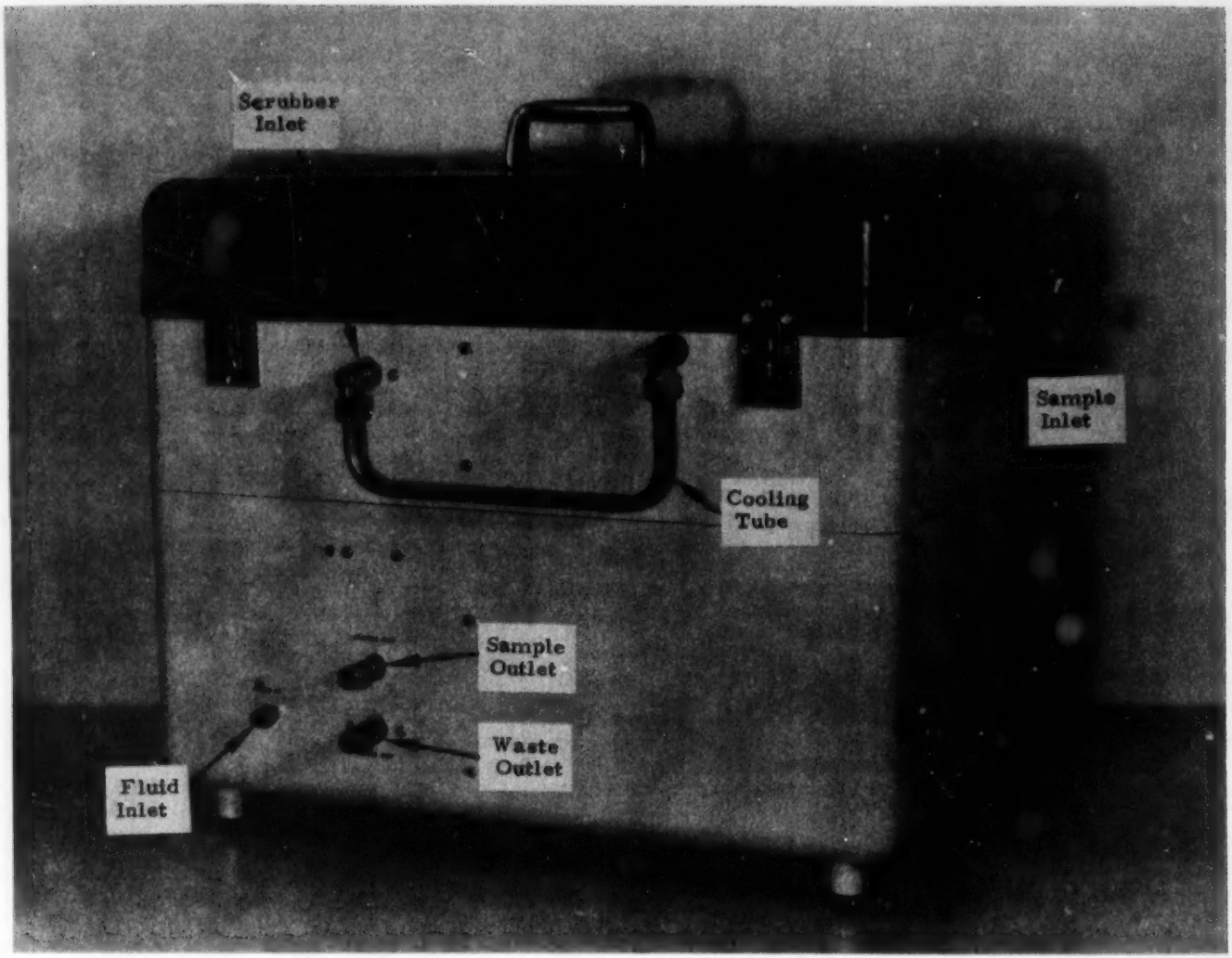


Figure A-2. Left Side View of Conditioner Showing

The Pyrometer Set Point Knob--

In the upper left corner of the front panel, there is a single set point pyrometer. This indicates the temperature of the furnace. The set point knob located in the bottom right corner of the pyrometer controls the temperature of the furnace. The furnace temperature is obtained by turning the knob until the red set point needle is at the desired temperature. The temperature will be maintained at that level.

The Fluid Pump Stroke Adjustment Knob--

The fluid pump stroke adjustment knob is located inside the case of the conditioner in the lower front left hand corner of the instrument, immediately behind the power terminal. It is reached by removing the cover of the unit. Release of the four trunk latches is required to remove the cover. This knob varies the stroke of the fluid pump which allows for different scrubber solution flow rates.

Plumbed Connections--

All of the plumbing connections to the conditioner are shown in Figure A-2. The Sample inlet for use when the furnace function is required is shown on the right side of the photograph. It is located on the back face of the unit. The cooling tube which passes the incoming gas from the furnace section to the scrubber is shown in the upper center. The left hand end of the cooling tube is connected to the lower inlet at the top of the glass scrubber and its coil. When thermal decomposition of the source sample is not required, the sample line from the probe or inlet should be connected to the scrubber inlet port. The hard-plumbed connection attached to the sample inlet (rear) may be moved to the scrubber inlet for this application.

At the lower left of Figure A-2 is the fluid inlet ("fluid in") connector. Inside the case, the fluid inlet is connected to the bottom side of the pump head. From the upper side of the pump head, the fluid line is connected to the upper most inlet of the glass scrubber coil by means of 4 mm (i.d.) teflon tubing. This connection is made with teflon tubing which is covered with teflon tape. Outside the case, the fluid inlet line passes to a 2 gallon heavy-duty polyethylene reservoir, which in operation will contain the scrubber solution. The connecting polyethylene tubing should reach to the bottom of the reservoir. It is sealed into a white plastic cap. This cap has only one tubing lead attached to it.

Next to the fluid inlet are two plumbed connectors, labelled "sample out" and "waste out".

From the "sample out" connector the scrubbed sample gas is removed from the conditioner unit. It passes to the diluter under usual operating conditions. A condenser and trap are connected to the "sample out" terminal to cool the gas stream prior to its entry into the diluter. Excess moisture is removed from the gas stream and collected in the glass trap by this technique. Figure A-3 shows the arrangement. Inside the module the "sample out" terminal is connected to the upper of the two takeoffs on the gas/liquid separator or trap located at the bottom of the scrubber coil. The connection is made with teflon tubing covered with teflon tape.

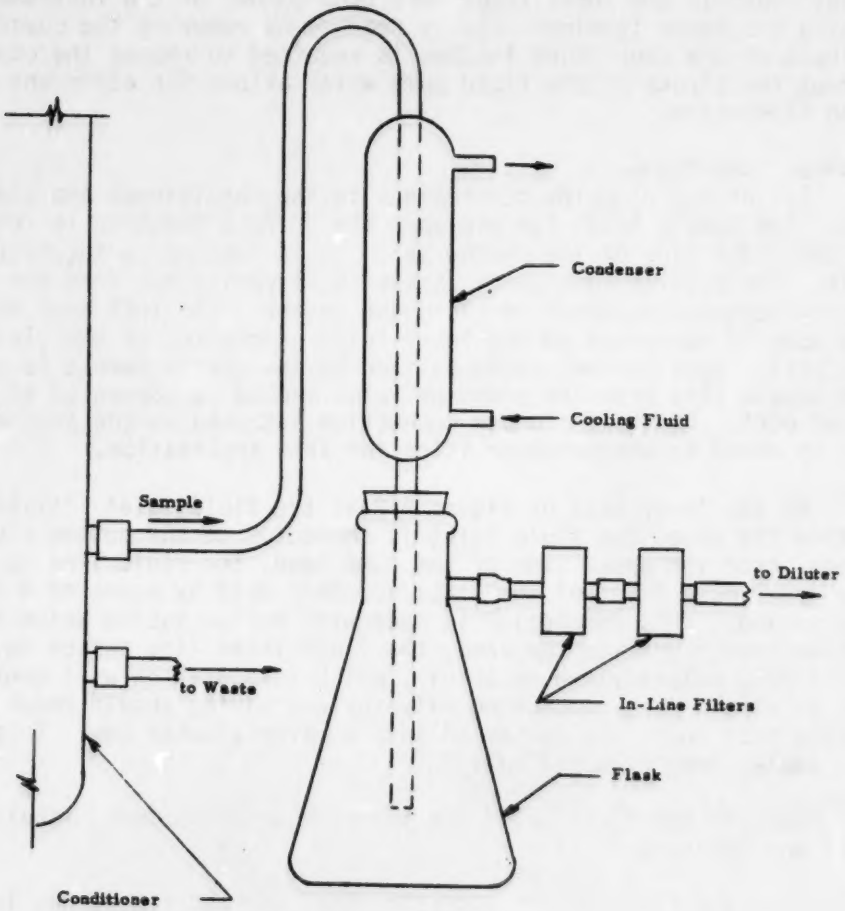


Figure A-3. Arrangement of condenser between conditioner and diluter modules.

Liquid waste from the scrubber leaves the unit from the "waste out" connector. Outside the case it connects to a two gallon container by means of 4 mm (i.d.) teflon tubing which is sealed into the top of a white plastic cap. The cap for the waste container has a second tubing lead extending from it which will be connected to the pump module. Within the module, the liquid waste line is connected to the bottom outlet from the trap (liquid/gas separator) located under the scrubber coil.

The Diluter Module

The Diluter Module has seven operating controls and three plumbed connections. Six of the controls shown in Figure A-4 are: the power switch, the dilution air switch, the low flow valve switch, the heater switch and two mass flowmeter set point knobs. The seventh control is the adjustable flowmeter which is shown in Figure A-5, a rear view of the diluter module. The three rear connections are the sample inlet, sample outlet, and the excess sample exhaust from the flowmeter.

The Power Switch--

Power to the unit is controlled by the red toggle switch on the right side of the front panel. In the "up" position, power is supplied to the solid state power supply and to the Rotron cooling fan. The light above the switch indicate when the power supply has been activated.

The Dilution Air Switch--

In the "up" position this white toggle switch initiates the sample dilution process. When activated the three way valve (shown in Figure A-3) closes off the sample line and allows the same flow rate of mercury free air to enter the unit through the filter of silver-alumina absorbent. This also activates both automatic servo valves for flow regulation.

In the "middle" position power is cut off to the automatic servo valves. If desired the valves may then be adjusted manually. This requires removal of the cover.

In the "down" position power is supplied to the high flow automatic servo valve. This position is used when the module is being used for flow regulation only.

The Low Flow Valve Switch--

This white toggle switch opens the two way valve in the sample line used during dilution (Figure A-3). It also activates the pump which maintains the sample gas flow through the lines and exhausts the excess gas. Both this switch and the dilution air switch must be "up" to have the sample gas diluted.

In the "down" position this switch shuts off the pump and closes the two way valve. This is the position used when no dilution of the sample gas is necessary. Both this switch and the dilution air switch must be in the "down" position when the unit is being used only for flow regulation and not dilution.

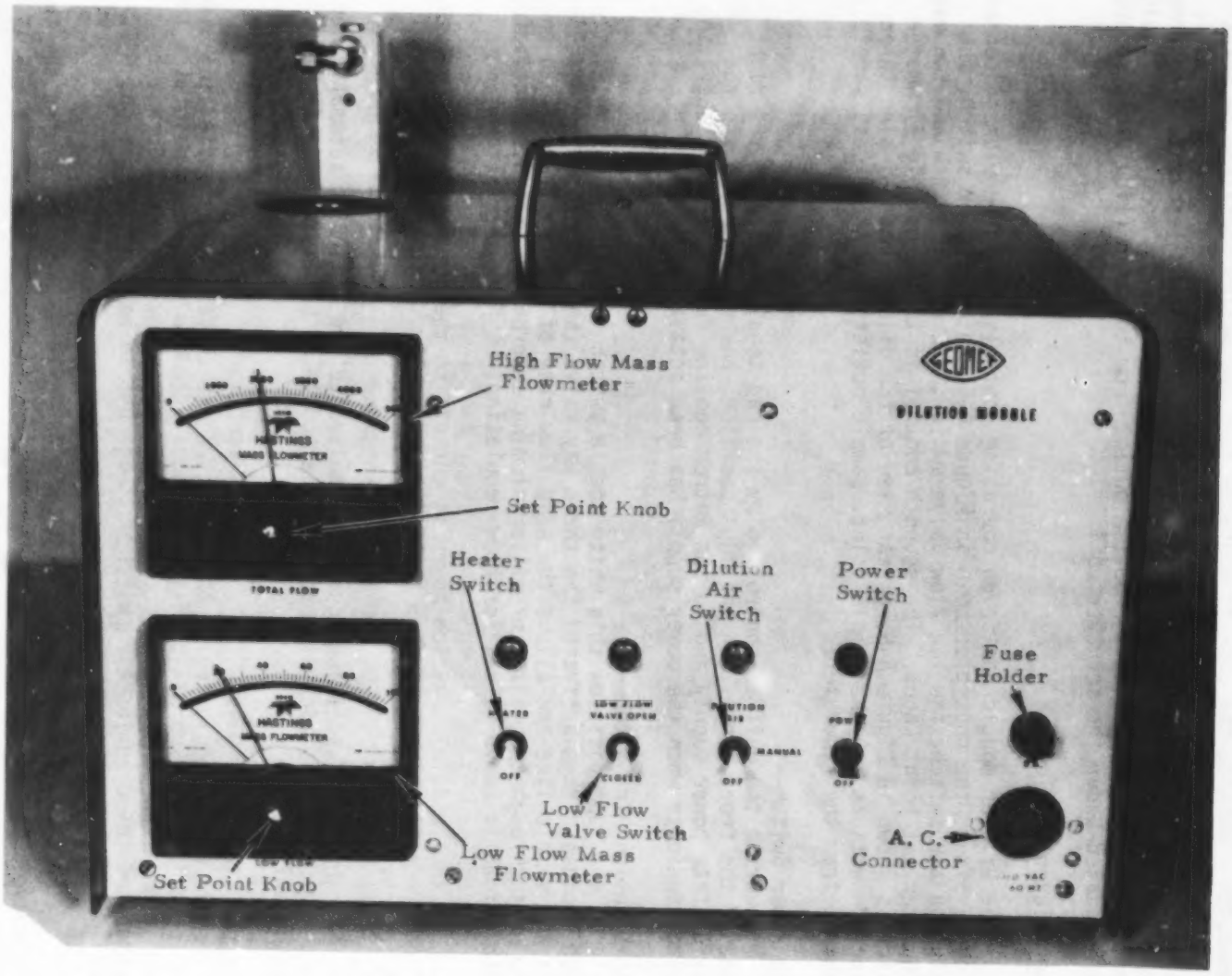


Figure A-4. Front Panel, Diluter Module

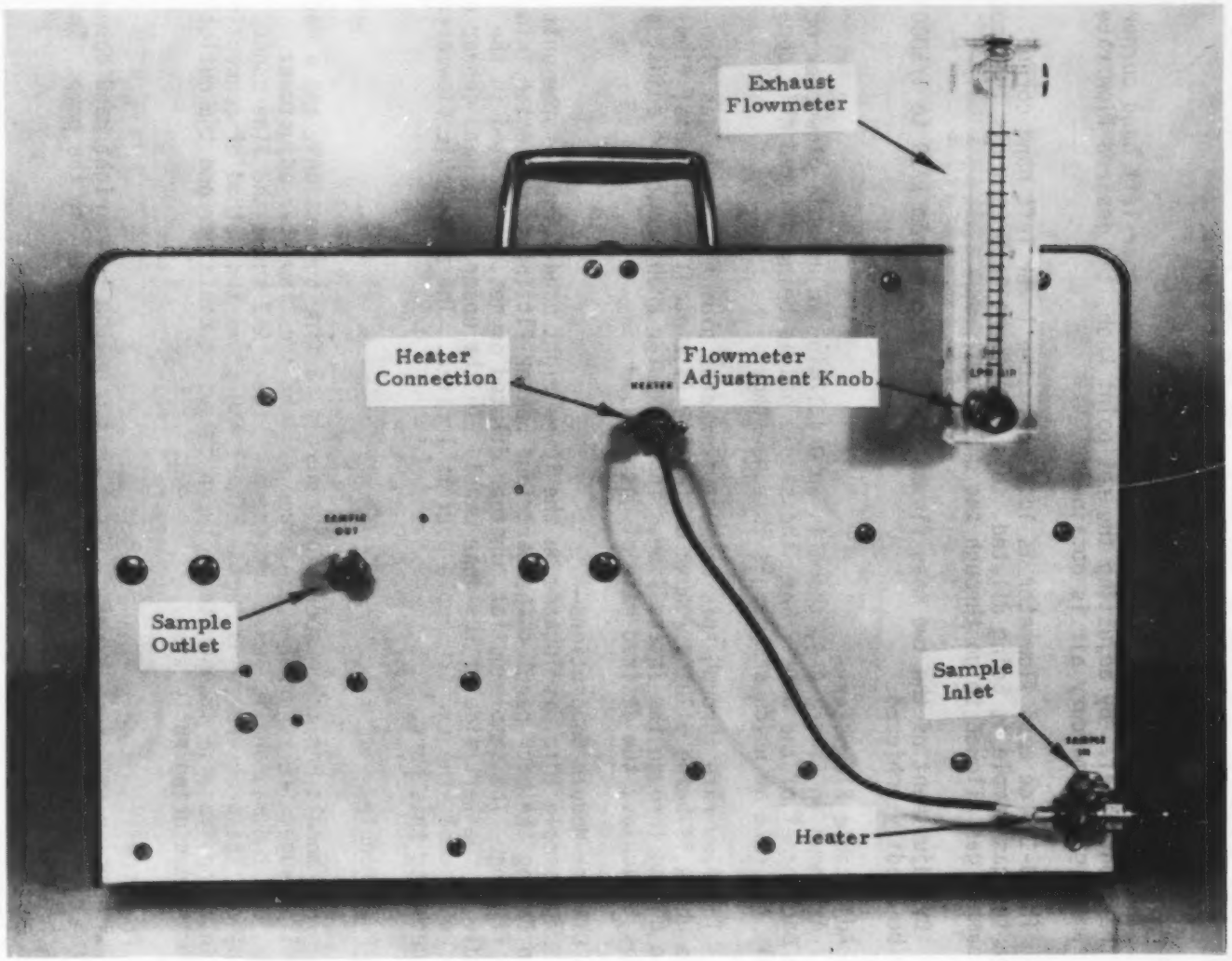


Figure A-5. Rear View, Diluter Module

The Heater Switch--

The white toggle switch which is labelled "heater" controls power to the cartridge heater on the back of the unit. In the "up" position the heater heats the incoming gas to prevent condensation in the unit.

The Mass Flowmeter Adjustment Knobs--

The 0-5 1/min. mass flowmeter is located in the upper left hand corner of the front panel. By adjusting the set point knob, the desired flow rate of sample gas or mercury air is obtained.

The 0-100 cc mass flowmeter is located in the lower left hand corner. When the gas sample is being diluted, this adjustment knob controls the amount of sample gas allowed to go through the unit.

By adjustment of the two mass flowmeters dilutions from 1/19 to 1/5000 may be readily achieved.

The Adjustable Flowmeter--

As shown in Figure A-4, there is an 0-4 1/min. adjustable flowmeter on the back panel of the unit. When the sample gas is being diluted this controls the rate at which sample gas is exhausted.

The flow rate through this exhaust flowmeter plus the sample gas flow rate which is being diluted (shown on the low flowmeter) is the total flow rate coming through the intake line. The flowmeter knob should be adjusted so as to achieve the desired rate of gas flow.

The Diluter Module Connections--

The sample inlet is located on the lower right hand corner of the unit when facing the back panel. The cartridge heater fitting is connected into the fitting. The sample outlet, whether diluted or not, is located in the middle of the left side of the rear panel. In the upper right hand corner of the rear panel, the excess sample port is located. The adjustable flowmeter connects to this port.

The Pump Module

This module has three operating controls, 3 line connections, and a magnehelic gauge. In Figure A-6 the power switch, the flowmeter adjustment knob, and the magnehelic gauge are shown. Figure A-7 shows the line connections and the pressure adjustment. Figure A-8 shows the method of connecting it into the system. It operates between the waste container and the outlet from the measuring photometer.

The Power Switch--

The power switch is a white toggle switch on the lower right hand corner of the front panel. In the "up" position this gives power to the pump. This switch is labelled "vacuum pump".

The Flowmeter Adjustment Knob--

In the middle of the front panel is a 0-0.5 1/min. flowmeter with an adjustment knob on the bottom. This regulates the gas flow rate through the

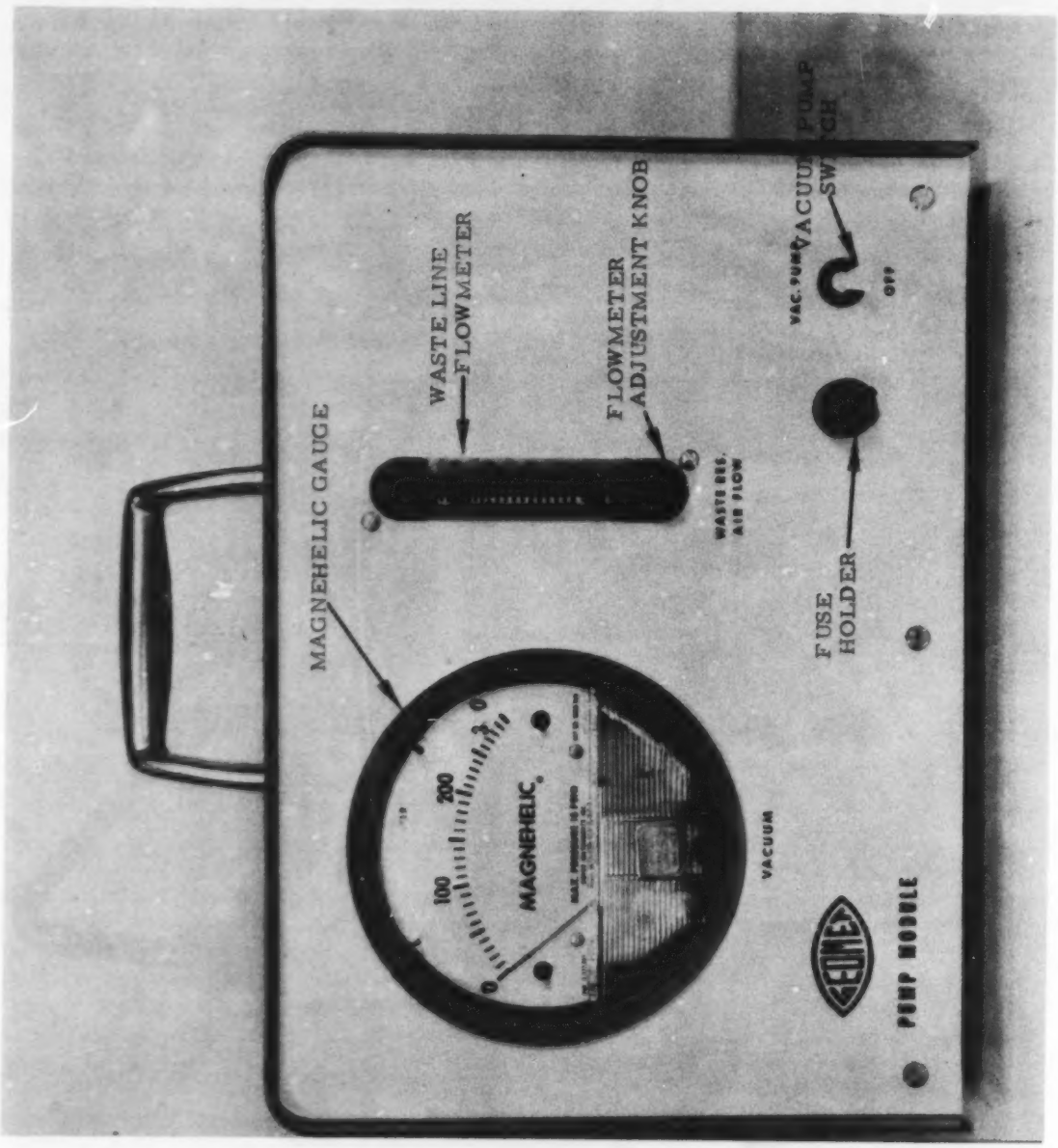


Figure A-6. Front panel, pump module.

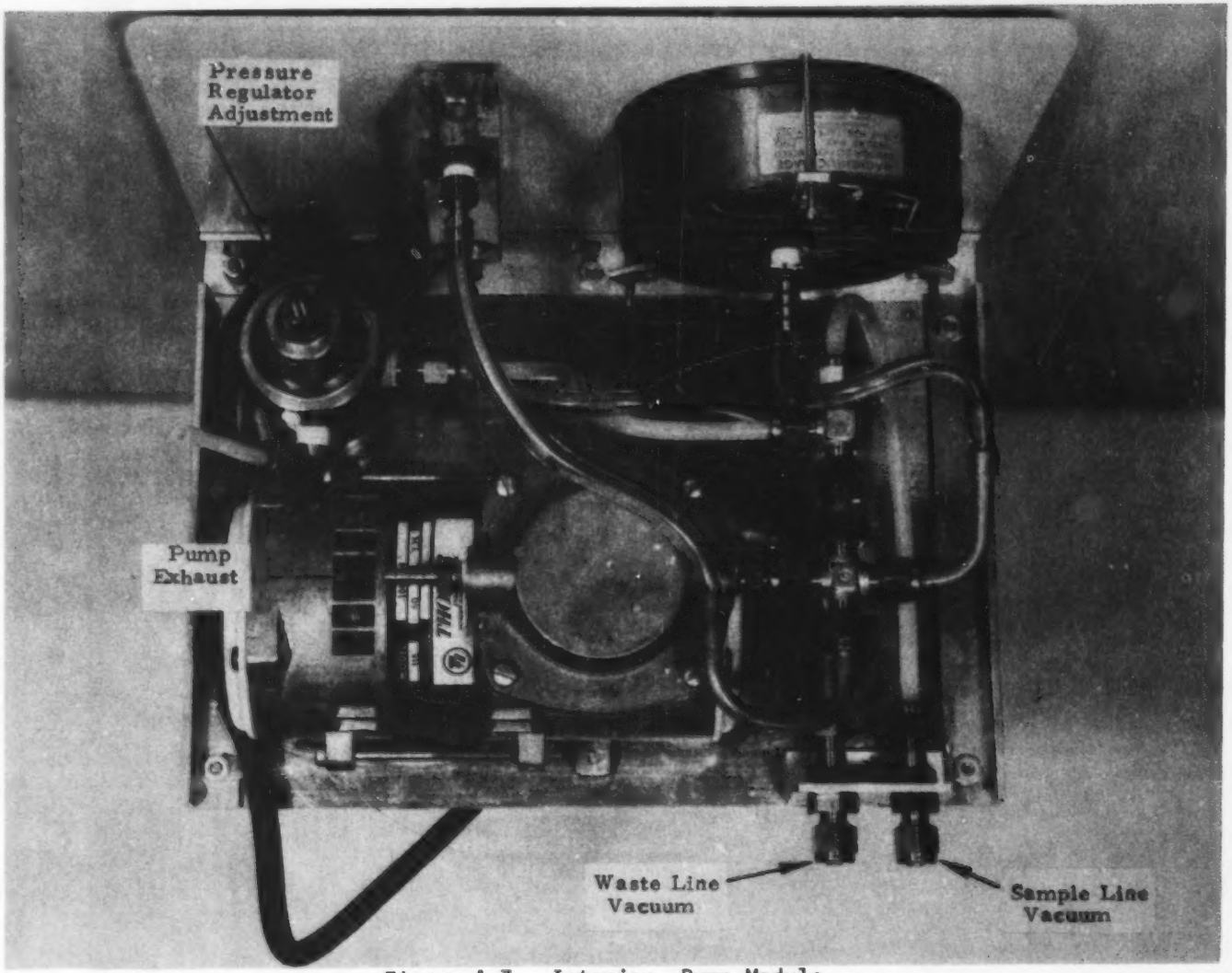


Figure A-7. Interior, Pump Module

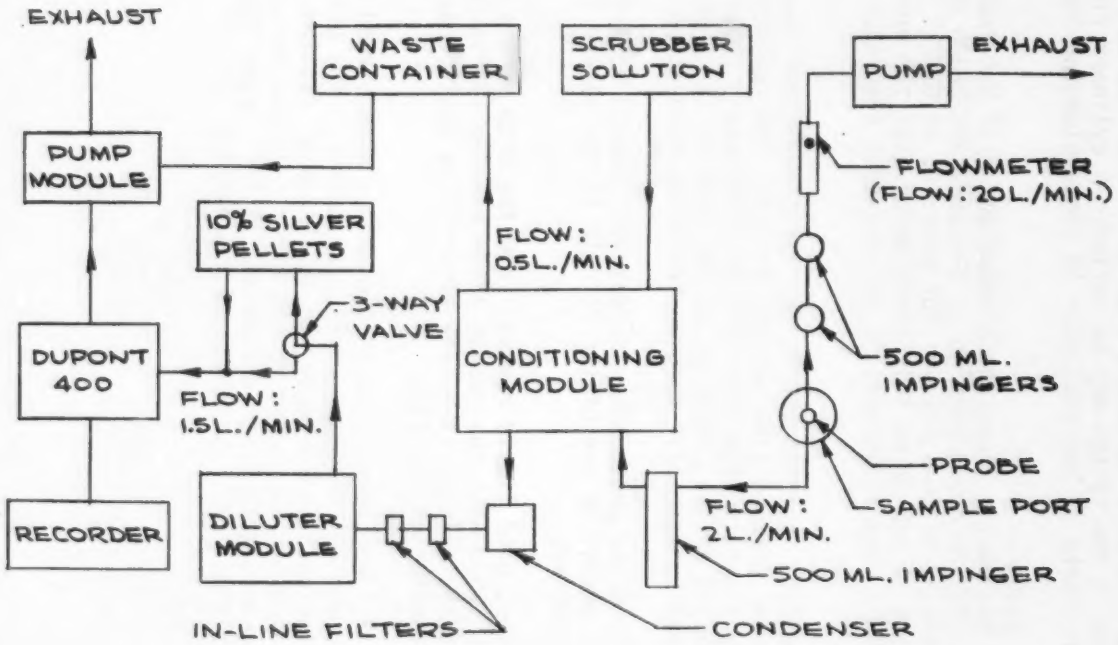


Figure A-8. Automated Mercury Interface system schematic.

waste line. Generally, the flow rate is maintained at 0.5 1/min.

The Pressure Regulator Adjustment--

Figure B-7 shows the interior arrangement of the pump module. The regulator which establishes and maintains a constant pressure inside the spectrophotometer cell is located inside this unit. It is in the lower front right hand corner when the unit is viewed from the front. By turning the threaded stem, the vacuum of the system may be varied. A cylinder filter is generally used on the air intake of the regulator to avoid plugging its orifice.

The Magnehelic Gauge--

The 0-300 Magnehelic gauge which reads in cm of H_2O indicates the vacuum in the system. The observed reading is the pressure below one atmosphere which is operational in the system. For example, at 0°C, 1 atm= 1033 cm of water. A reading of 100 cm on this gauge means that the pressure in the system is 933 cm H_2O. Any adjustment of the pressure regulator can be observed on this gauge.

The pump module is connected to the waste container (waste line vacuum) and the photometer (sample line vacuum) outlet. The connection to the waste line is made by inserting the 1/8" line from the waste container to a 1/4" polyethylene busing at the connector at the rear of the unit. Tightening the teflon ferrule on the 1/4" bushing seals the connection.

The line from the photometer (DuPont 400) should be 1/4" polyethylene tubing which is sealed by use of the ferrule at the sample line fitting (right hand connector).

In operation, good liquid removal from the trap at the bottom of the scrubber was achieved with the vacuum gauge reading 225 inches of water. This included use of the diluter as a flow controlling monitor at 2.0 1/min. and the DuPont Photometer in the line.

There are 3 line connections of the unit. When viewed from the rear, two connections are on the lower right hand corner. The outlet line from the spectrophotometer cell is connected to the "sample" fitting. The line from the waste container is connected to the other. Inside the unit on top of the pump, the pump exhaust fitting is located. A 1/4" tygon tube may be connected to the exhaust if desired. Normally it is operated "as is".

SECTION A2

REAGENTS AND INSTRUMENT PREPARATION

Once the units have been brought to a test site, they must be readied for use. This entails preparing the reagents, installing glassware in the conditioning module, and making the appropriate connections for the gas and liquid flow systems. Figure A-8, a schematic diagram shows the interface system connected and ready to use.

Reagents

For the operation and calibration of the system, five solutions are needed. These are the scrubber solution, the stannous chloride solution, and the sodium borohyride solution.

The Scrubber Solution--

The scrubber solution is 20% 0.2M ammonium bicarbonate (NH_4HCO_3), 80% 0.2M sodium bicarbonate ($NaHCO_3$), to which 2 ml of 10% Triton X100 per liter has been added. To make a two gallon supply, 20 g of reagent grade ammonium bicarbonate, 102 g of commercial sodium bicarbonate, and 15 ml of 10% Triton X100 are added to two gallons of distilled water. After thoroughly mixing the solution, it is ready for use.

The Saturated Sodium Bicarbonate Solution--

The two impingers shown in Figure A-8 are partially filled with this solution. It is made by placing 30 g of sodium bicarbonate in each impinger with 100 ml of water. This produces a saturated solution with excess solid remaining to be dissolved as the sodium bicarbonate reacts with sulfur dioxide.

The Acid-Permanganate Solution--

This solution is used in some of the calibration procedures to collect mercury. It is generally prepared in 15 ml amounts which is the amount held by a bubbler. To make 15 ml, 1.0 ml of 0.25M potassium permanganate and 2.5 ml of 1:1 nitric acid are diluted to 15 ml with distilled water. This is used when the gas stream contains oxidants. When transferring collected mercury from one bubbler to another, only 0.3 nl of permanganate are used with the acid and water.

The Stannous Chloride Solution--

The stannous chloride solution is used to reduce the acid-permanganate solution during analysis. The solution is 20% W/V stannous chloride in 6N hydrochloric acid. To 100 ml of 6N HCl, 20 g of reagent grade stannous chloride is added. After thoroughly mixing the solution, it is ready to use.

The Sodium Borohydride Solution--

This is used in conjunction with the analysis of the mercury collection bubblers. It is used in a bubbler which is connected to the bubbler to be analyzed. For a full day of analyses, only 15 ml or a convenient level in the bubbler is needed. It is prepared by adding 1 g of sodium borohydride ($NaBH_4$) to 15 ml of distilled water.

The Conditioning Module-Installation of Glassware

The Conditioning Module is prepared for use by the installation of the glassware and by making the appropriate connections. The unit comes with two pieces of glassware which are to be fastened in with ball and socket clamps. Inside on the side of the unit there are three 12/5 socket joints. The waste separator is clipped to the bottom two. The bottom ball joint of the scrubber coil is clipped to the waste separator. The top socket on the unit wall is clipped to the ball joint of the scrubber coil. The other ball joint on the top of the coil is connected to the socket joint on the solution line from the pump. Figure A-9 shows the unit with the glassware installed.

Gas Flow System

As shown in Figure A-8, the interface apparatus is connected to the probe with a glass tee. The tee has portions of three ball joints: a 28/15 ball, a 28/15 socket, and a 12/5 ball. It is used to divide the gas flow between the interface sample line and an exhaust line. To obtain a reliable sample, more gas than is used by the interface system is drawn through the probe, and the surplus is exhausted. For simultaneous comparisons, the exhaust system is composed of two Greenburg-Smith impingers, a 36 1/min. flowmeter, and a ~30 1/min. Thomas Pump. These are connected by 1/2" I.D. rubber tubing and ground glass joints.

The sample intake line to the interface instrumentation which has a 12/5 socket joint to connect to the glass tee is 30" of 3/16" I.D. amber latex tubing which connects to an empty Greenburg-Smith impinger. This in turn is connected to the sample inlet of the conditioning module by another 30" of amber latex tubing.

In the conditioning module the gas flow is split. Most of the sample exits the "sample out" connection through a short section of teflon tubing to the condenser and thence to the diluter and photometer. However, approximately 0.5 1/min. of it travels through the "waste out" connection to the waste container along with the waste scrubber fluid. The waste and fluid reservoir containers are fitted with teflon tubing (1/8" O.D., 1/16" I.D.). From the waste container this 0.5 1/min. gas flow is drawn to the pump unit where the flow is regulated and then exhausted.

The condenser between the conditioner and diluter modules is connected by means of ground joints to condensate collection flask. (Figure A-3). The gas passes through the side arm of the flask to the two heated 25 mm Gelman filter holders which are connected by short sections of amber latex tubing. A heating tape connected to a variac heats the filter holders and eliminate any problems with water on the Whatman #41 filter paper. These

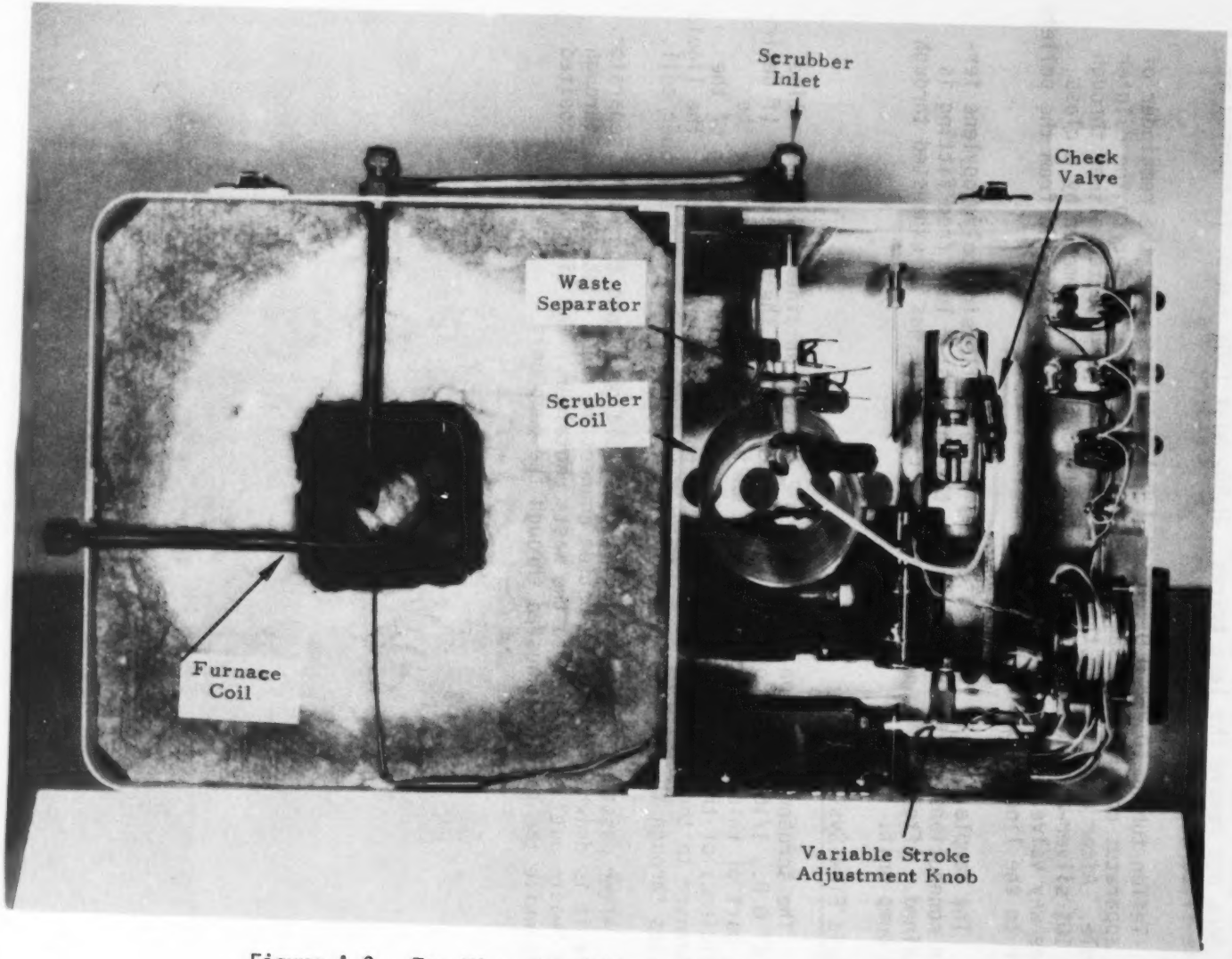


Figure A-9. Top View, Conditioner Module-Cover Removed

filters guard against passage of any residual fly-ash, in the event the scrubber does not remove the last traces. H_2SO_4 mist ($SO_3 + H_2O$) is removed at these filters also.

Teflon tubing (0.25" O.D., 0.19" I.D.) which connects the remainder of the apparatus conducts the sample gas from the filter holder to the diluter module. After exiting from the diluter, the gas is directed either through the 10% silver-alumina pellets or to the DuPont 400 photometer by a glass three way valve. A polypropylene Y serves to connect the line from the pellets back to the line to the photometer.

The sample line is fastened to the teflon cell using polypropylene ferrule connections. With teflon tape on the threads, a leak free fitting is obtained. After exiting from the monitor cell, the gas is exhausted through the pump unit.

Liquid Flow System

The scrubber solution is held in a 2 gallon container. A teflon line (1/8" O.D., 1/16" I.D.) connects it to the bottom of the condenser. If desired, part of this line may be placed in an ice bath so as to improve the efficiency of the condenser. The fluid then travels out of the top of the condenser to the "fluid in" connection of the conditioning module. The liquid passes through the pump and a check valve before entering the scrubber coil.

After passing through the coil, the fluid goes into the waste separator. Here it is drawn off from the bottom connection along with some gas through the "waste out" connection to the waste container. The liquid is deposited here while the gas is exhausted through the pump unit.

SECTION A3

INSTRUMENT OPERATION

The Automated Mercury Interface hardware is flexible and can be used in a variety of ways. With the modular composition of the apparatus, it can be adapted to meet various testing situations. The conditioning module which prepares the gas sample for photometric measurements and the diluter module which regulates and or dilutes the sample may be interchanged or in some cases omitted to meet different testing requirements. Descriptions will be given of each of the operating modes.

Non-Flammable Sources

In testing sources such as power plants, there is no danger of sampling flammable gases. The following sections will indicate different methods of operating the interface system with non-flammable sources.

Complete Interface System--

When testing a source with non-flammable gas, the system may be set up to operate as in Figure A-8. After connecting the apparatus, it is necessary to establish the proper gas flow rates.

The middle two switches of the diluter module are initially kept in the down position. With the sample line unhooked from the probe tee, the 0-5 1/min. mass flowmeter is adjusted to 1.5 1/min. or to any other desired setting. The flowmeter on the pump module is adjusted to draw 0.5 1/min. through the waste line. This makes the flowrate 2.0 1/min. through the sample line at the probe. With 100 ml of saturated sodium bicarbonate in the impingers, the flowrate of the exhausted excess gas drawn through the probe is adjusted to ~20 1/min.

The scrubber solution container is then filled, and a check is made of the solution flowrate into the scrubber. This is done by unhooking the line from the top of the scrubber coil and placing it in a graduated cylinder. The flowrate is determined from the volume of collected liquid, and the length of time during which the collection occured. A flowrate of 5 ml/min. is sufficient for most applications. If the flowrate is unacceptable, it may be adjusted using the "Fluid Pump Stoke Adjustment Knob". Once the flowrate is correct, it should not need to be changed during the test.

A 25 mm circle of Whatman #41 filter paper is placed in each of the filter holders. These need to be replaced about every two hours, when sources having high paritcle loadings are monitored. However, they last much longer depending on the amount of flyash present. The filter holder are kept warm to

the touch by the heating tape which is controlled by a variac. If the variac is set at approximately 5 divisions, the heating tape should provide enough heat.

The temperature of the conditioning module furnace is generally set at 300°C. This may be varied depending on the testing circumstances. However, 300°C was found to be sufficient for most applications.

With the sample inlet line to the conditioner still unhooked at the probe tee, a base line is obtained on the 10 mv scale of the recorder. After checking to be sure that all gas and liquid flows are proper, the inlet line is clipped to the probe tee. Once a steady signal has been achieved, the three way valve is turned to divert the gas through the silver-alumina tablets. The signal difference indicates the mercury contribution to the signal.

If the signal goes back to the base line after insertion of the tablets, the entire signal can be attributed to mercury. However, if it does not, it is an indication that a nonmercury interference is also giving a signal. The calibration of these two cases will be discussed in a later section.

It may occur that a very small signal or no signal will be apparent on the 10 mv scale. In this case the 2 mv scale should be used. The contribution of the mercury to the signal may be determined as before.

If a very high signal is obtained, dilution of the sample will be required. The middle two switches on the diluter module front panel are placed in the "up" position. The set point knob of the low flow mass flowmeter is then turned to yield a flowrate which gives an acceptable signal. The rotameter on the back panel of the diluter module must be adjusted to give approximately the same flowrate through the sample line as before.

The apparatus between the probe and the conditioning module should be cleaned daily. This involves rinsing the lines and impingers with distilled water. The probe should be checked periodically and cleaned if necessary. The pressure regulator in the pump unit should have a filter on the air intake. If a 4" long, 1" I.D. cylindrical filter is used, it will last over a week in normal operation.*

Operation Without the Diluter Module--

It may be convenient on occassion to remove the Diluter Module from the system. The resulting interface apparatus will consist of the Conditioner in such operation. In these situations, the Diluter Module is replaced by a flowmeter and a glass valve. With this arrangement the operational procedure indicated previously is followed with the following exception:

A periodic check of the air flow of the sample line at the probe tee should be made. This is necessary because the vacuum of the system cause an

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* A cartridge filter such as the Gelman, Pleated Membrane Capsule, Cat. No. 12104 may be connected by use of a short section of rubber tubing.

error in the flowmeter which replaces the diluter. This flowmeter and the valve also may be heated to avoid condensation if the water content of the sample if high.

Bypassing the Furnace--

It is possibe that the furnace of the conditioning module may not be necessary in some tests. If mercury compounds are not present or are not of interest, the furnace may be bypassed. The sample gas then goes directly to the scrubber coil.

This is done by first removing the line from the sample inlet of the module. The fitted sample inlet tube may be connected at the side port entry to the scrubber, marked scrubber inlet in Figure A-9. Then, the sample from the probe bypasses the furnace and enters the scrubber directly. The furnace heater switch is kept in the "down" position when operating in this mode. Otherwise, the operating procedures remain the same.

Flammable Sources

In many plants the gas streams contain hydrogen or other flammable substances. To test these sources requires that the sample be diluted prior to entering the furnace. This mode of operation is shown schematically in Figure A-10. Operating procedures remain essentially the same with an important exception.

Flammable sources require that the gas be diluted below the flammability threshold. This is accomplished by changing the order of use of the modules: The diluter precedes the conditioner. A filter will probably be necessary in front of the diluter module to avoid clogging the mass flowmeter transducer.

If the mercury level of the source is high, this mode of operation presents no difficulties. However, with low mercury levels, the diluted sample may not contain a measurable level of mercury. In this situation it may be useful to operate as in the preceding section where the furnace is bypassed.

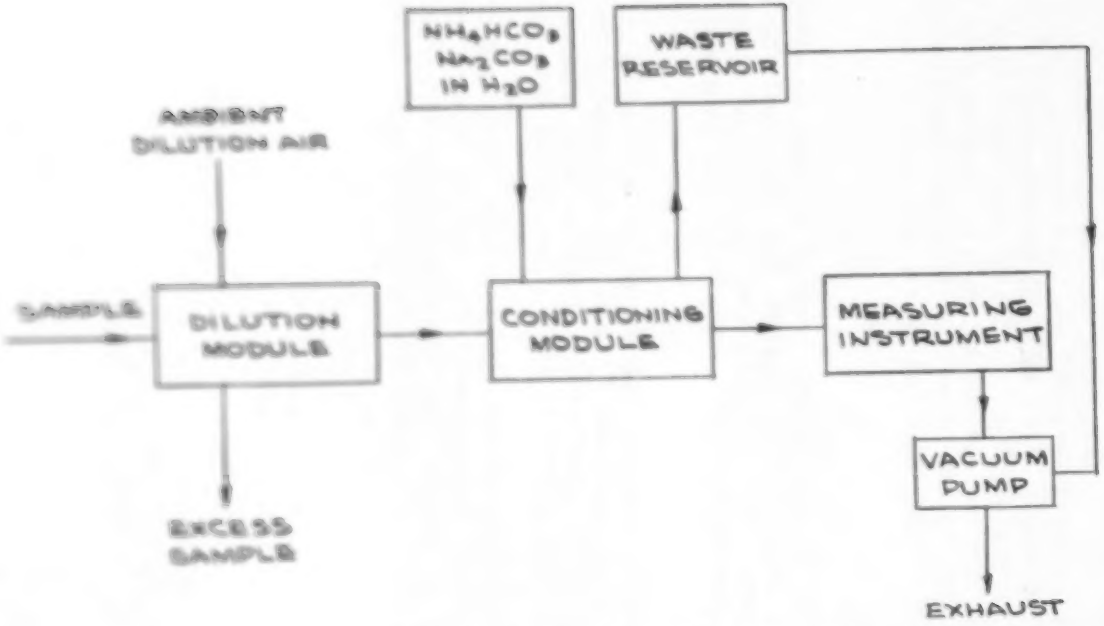

Figure A-10. Use of Interface System with flammable substances.

SECTION A4

CALIBRATION

Calibration of the instrument is partly contingent on the type of signal which is obtained. Two methods of calibration will be detailed which should cover all situations. The calculations necessary to obtain the mercury concentration in the gas stream will also be given.

Calibration of the Spectrophotometer in the Laboratory

Before going into the field, a calibration curve for the photometer installed in the operating system should be obtained in the laboratory. If it is found to be valid in the test situation, it is the easiest method of determining the mercury content of the stack gas. This first method requires mercury levels over $\sim$10 $\mu g/m^3$ with little or no signal interferences.

To obtain a calibration curve similar to that of Figure A-11, it is necessary to have a system for generating various steady levels of mercury vapor. Such a system is shown in Figure A-12. The photometer response to a constant mercury level is obtained, and this level is then quantitated with acid-permanganate bubblers which are prepared as described in Section 2.

To quantitate the mercury level, a calibration curve for the acid-permanganate bubblers must be obtained first. Known amounts of mercury are obtained from a source such as in Figure A-13, which is used in conjunction with the data in Table A-1. Various volumetric vapor samples are collected in bubblers, and each bubbler is then analyzed to give a point on the calibration curve.

To analyze the bubbler, an atomic absorption spectrophotometer is usually used. A pump is used to draw 2 l/min. of clean air through the spectrophotometer cell and a sodium borohydride bubbler (described in Section A-2), the contents of the mercury-containing bubbler is then reduced with 2.0 ml of stannous chloride solution (Section A-2), shaken vigorously and connected to the borohydride bubbler. The resulting peak height is measured, and a calibration curve is plotted. This allows quantitation of the mercury level which in turn established the calibration of the spectrophotometer.

If the photometer calibration is not carried out using the full interface system, it is necessary to correct for the vacuum in the photometer cell. This correction can be built into the curve if the system is always run at the same vacuum, or it can be calculated. The manometric gauge on the Pump Module serves to indicate the cell vacuum.

If there are no signal interferences during testing, the method described above is the easiest for determining the mercury content of the sample gas.

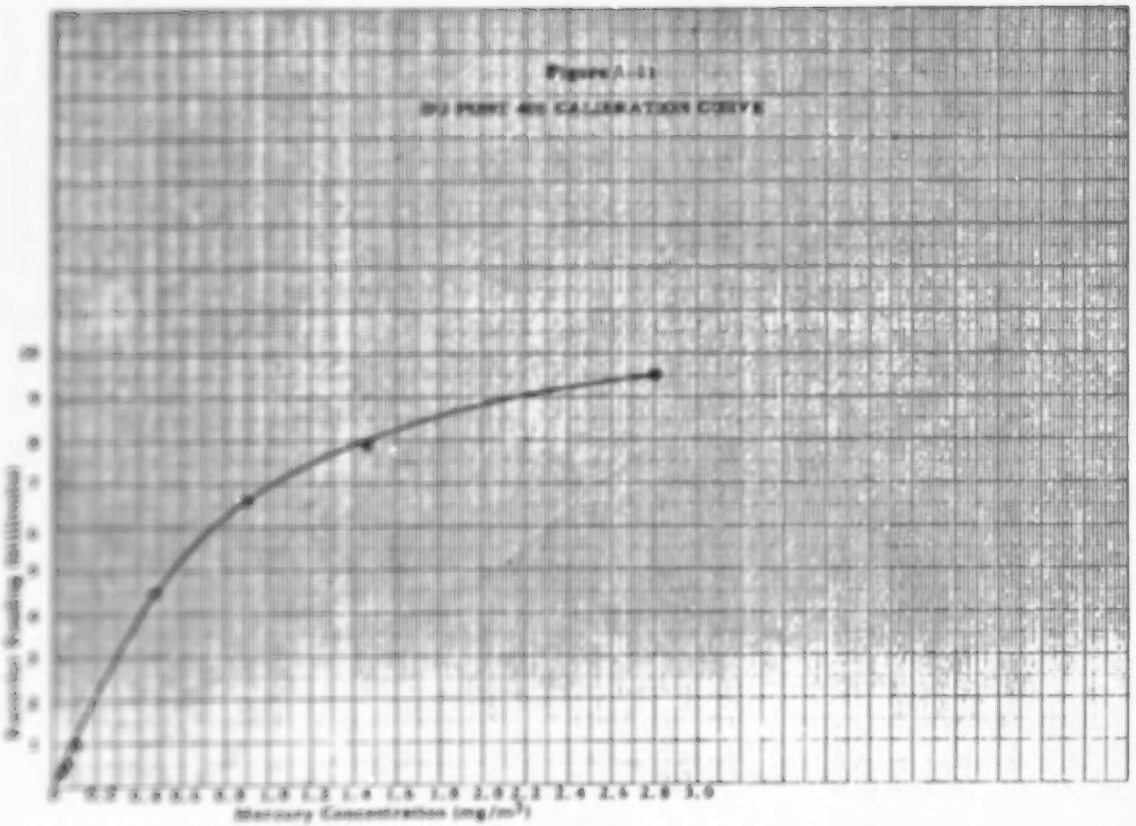

Figure A-11. Du Pont 400 calibration curve.

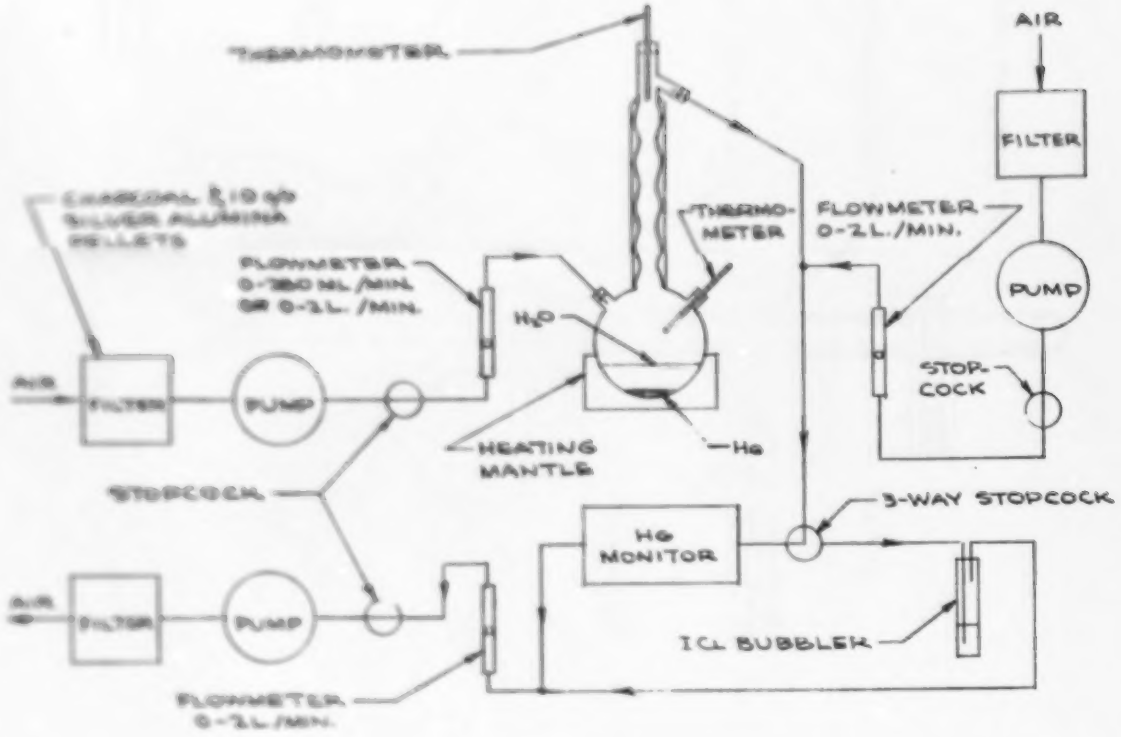

Figure A-12. Mercury vapor generating system.

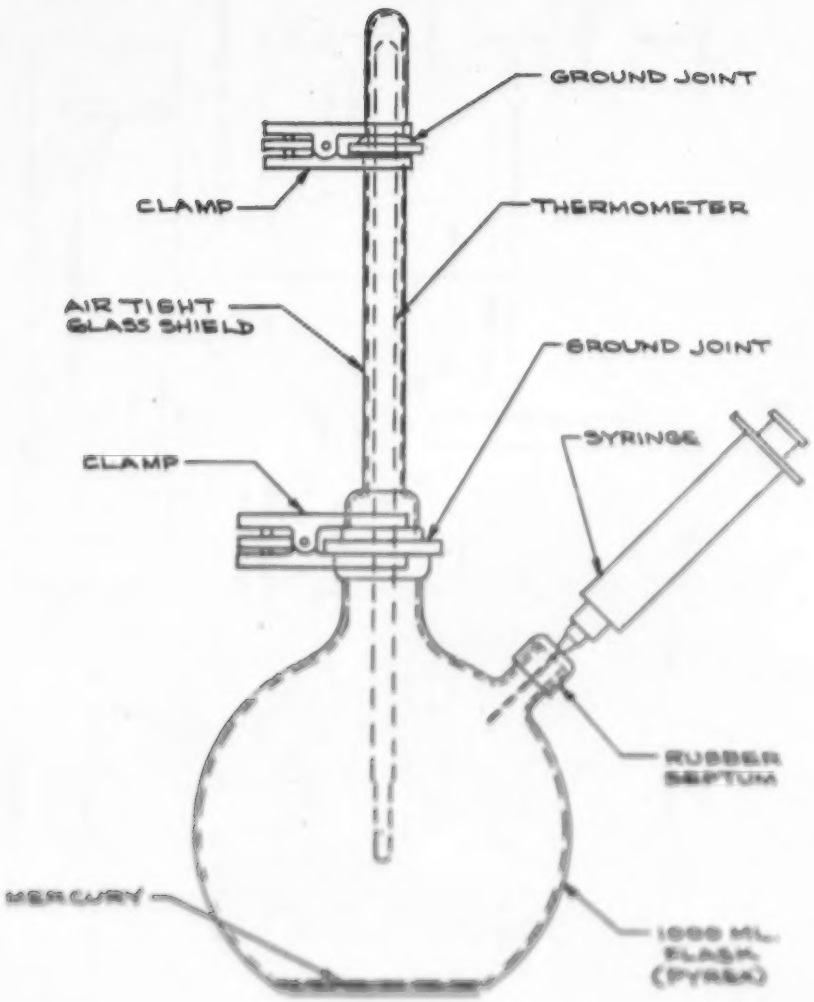

Figure A-12. Mercury reservoir flask for withdrawing mercury vapor samples.

TABLE A-1 MERCURY CONTENT OF SATURATED VAPOR

Temperature - Volume Relationships of Mercury Vapor

Temp(°C)	ng/cc	cc/100ng	Temp(°C)	ng/cc	cc/100ng
19.0	12.0	8.34	23.0	16.8	5.96
19.1	12.1	8.26	23.1	17.0	5.88
19.2	12.2	8.20	23.2	17.1	5.85
19.3	12.3	8.13	23.3	17.2	5.82
19.4	12.4	8.06	23.4	17.4	5.75
19.5	12.5	8.00	23.5	17.5	5.72
19.6	12.6	7.94	23.6	17.7	5.65
19.7	12.7	7.87	23.7	17.9	5.59
19.8	12.8	7.81	23.8	18.0	5.56
19.9	12.9	7.75	23.9	18.2	5.50
20.0	13.0	7.70	24.0	18.3	5.46
20.1	13.2	7.58	24.1	18.5	5.40
20.2	13.3	7.52	24.2	18.6	5.37
20.3	13.4	7.46	24.3	18.8	5.32
20.4	13.5	7.40	24.4	18.9	5.30
20.5	13.6	7.35	24.5	19.1	5.24
20.6	13.7	7.30	24.6	19.2	5.21
20.7	13.9	7.20	24.7	19.5	5.13
20.8	14.0	7.15	24.8	19.7	5.07
20.9	14.1	7.10	24.9	19.9	5.03
21.0	14.2	7.04	25.0	20.0	5.00
21.1	14.3	7.00	25.1	20.1	4.98
21.2	14.5	6.90	25.2	20.3	4.93

(Continued)

TABLE A1(continued)

Temperature - Volume Relationships of Mercury Vapor

Temp(°C)	ng/cc	cc/100ng	Temp(°C)	ng/cc	cc/100ng
21.3	14.6	6.85	25.3	20.5	4.88
21.4	14.7	6.80	25.4	20.6	4.85
21.5	14.8	6.75	25.5	20.8	4.81
21.6	15.0	6.67	25.6	21.0	4.76
21.7	15.1	6.63	25.7	21.2	4.71
21.8	15.2	6.59	25.8	21.4	4.67
21.9	15.3	6.54	25.9	21.5	4.65
22.0	15.5	6.45	26.0	21.7	4.61
22.1	15.6	6.41	26.1	21.9	4.57
22.2	15.7	6.37	26.2	22.0	4.54
22.3	15.8	6.33	26.3	22.2	4.50
22.4	16.0	6.25	26.4	22.3	4.48
22.5	16.1	6.21	26.5	22.5	4.44
22.6	16.3	6.14	26.6	22.8	4.39
22.7	16.4	6.10	26.7	23.0	4.35
22.8	16.5	6.06	26.8	23.2	4.31
22.9	16.6	6.02	26.9	23.4	4.27

The recorded signal height from the gas can be converted directly into a mercury concentration using the spectrophotometer calibration curve. However, if interferences are present, this method probably will not be useful. Despite the use of the optical filter system in the photometer, small interferences occasionally appear. In the presence of low levels of mercury vapor these may be significant.

Dynamic Calibration

If interferences cause the spectrophotometer curve to be unuseable, calibration in the field may be readily carried out.

When an interference is present, the 10% silver pellets may be used to establish the mercury levels. The pellets quantitatively absorb mercury, and when the sample gas passes through them, the photometer gives a signal which defines the nonmercury interference. The difference between this signal and the signal of the mercury plus the interference is the mercury signal. The calibration of this mercury signal is then necessary.

This is accomplished using a mercury source such as in Figure A-13. A known amount of mercury vapor (see Table A-1) is injected into the sample line at the probe tee, and with a chart speed of 0.1"/sec., a large peak is obtained. This peak is then used to quantitate the value of the mercury content of the sample gas.

The area of the peak is calculated in chart paper squares (or by use of an integrator or by weighing) and a rectangle represents a constant level of mercury over the period of time that it took to form the peak. The mercury concentration represented by this level is then calculated from the amount of mercury which formed the peak and the volume of gas which carried the peak through the system. The volume is based on the flow calibration established previously.

The average value of the mercury concentration in the sample gas is obtained as a result. Since the signal area is directly proportional to the mercury content, comparisons of signals from known mercury levels obtained in this fashion with signals from test levels will yield the value of unknown mercury concentrations.

To obtain the best results with this mercury source, it should be placed in a water bath or at least insulated from rapid temperature change. The thermometer used in the source usually has a limited range. This may present problems if the area where the equipment is set up is subject to rapid temperature change. A constant temperature water bath would be most useful if this problem is anticipated.

SECTION A5

MAINTENANCE

Some maintenance other than the routine cleaning of lines and impingers is required on each of the three modules. The maintencance required is not difficult and does not require much time.

The Conditioning Module

Maintenance of this module concerns cleaning of the furnace and scrubber coils. Both coils can be removed from the unit to be cleaned.

To remove the furnace coil, the top layers of insulation are first removed to expose the top of the coil. Both ends of the coil are then pushed back through the instrument wall. The coil is then lifted out. A water rinse followed by blowing out with an air hose is generally sufficient to clean the coil.

Depending on the particulate level, it is anticipated that after a week of operation, the scrubber coil may require cleaning. To do this the coil is removed from the unit. Pass some of the stannous chloride hydrochloric acid solution through the coil. This solution has proven to be a useful cleaning agent in the field. In the event that deposits are difficult to remove, a variety of other cleaning or oxidizing agents may be applied. Rinse carefully prior to reinstallation in the Conditioner.

Diluter Module

Water in the transducer of the mass flowmeters is an occasional maintenance problem with the diluter module. The symptoms of the problem are erratic or zero air flow indications by the meter when gas is passing through the unit. The problem is solved by removal and drying of the transducer.

First, disconnect the power supply from the unit. To remove the transducer, the cover must be taken off the diluter instrument. This exposes the inside of the unit which is shown in Figure A-14. The spacer bar is removed, and the wires are disconnected from the circuit board. After disconnecting the cable from the transducer, the module is effectively divided into front and back halves. The front half may then be removed by removing the four screws at the bottom of the front panel. This will expose the rear panel as shown in Figure A-15. The transducer is then removed by disconnecting the fittings on both ends and by removing the two large screws which hold it to the back panel.

Once it is removed, the transducer should be rinsed with acetone. The

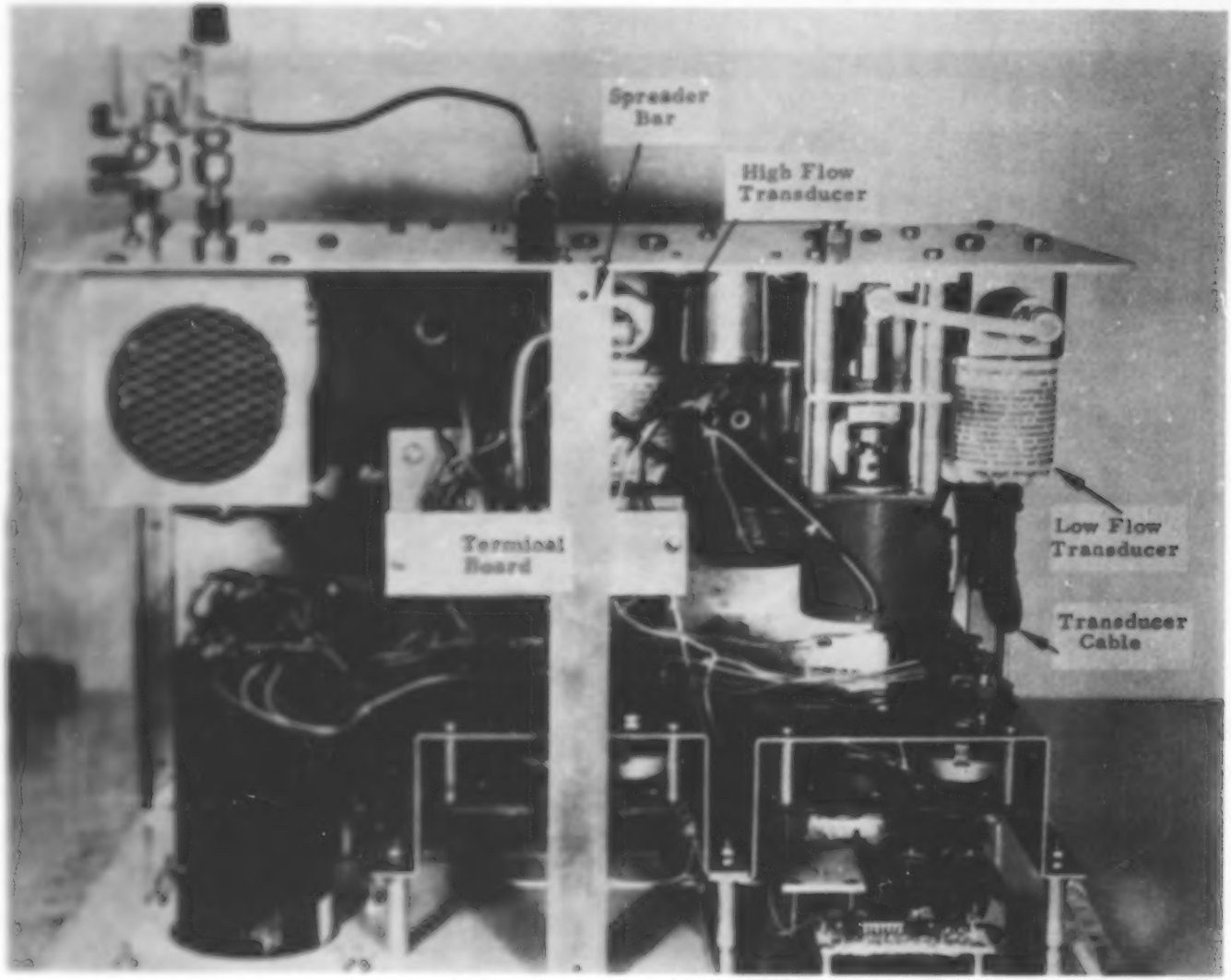

Figure A-14. Top View, Diluter Module

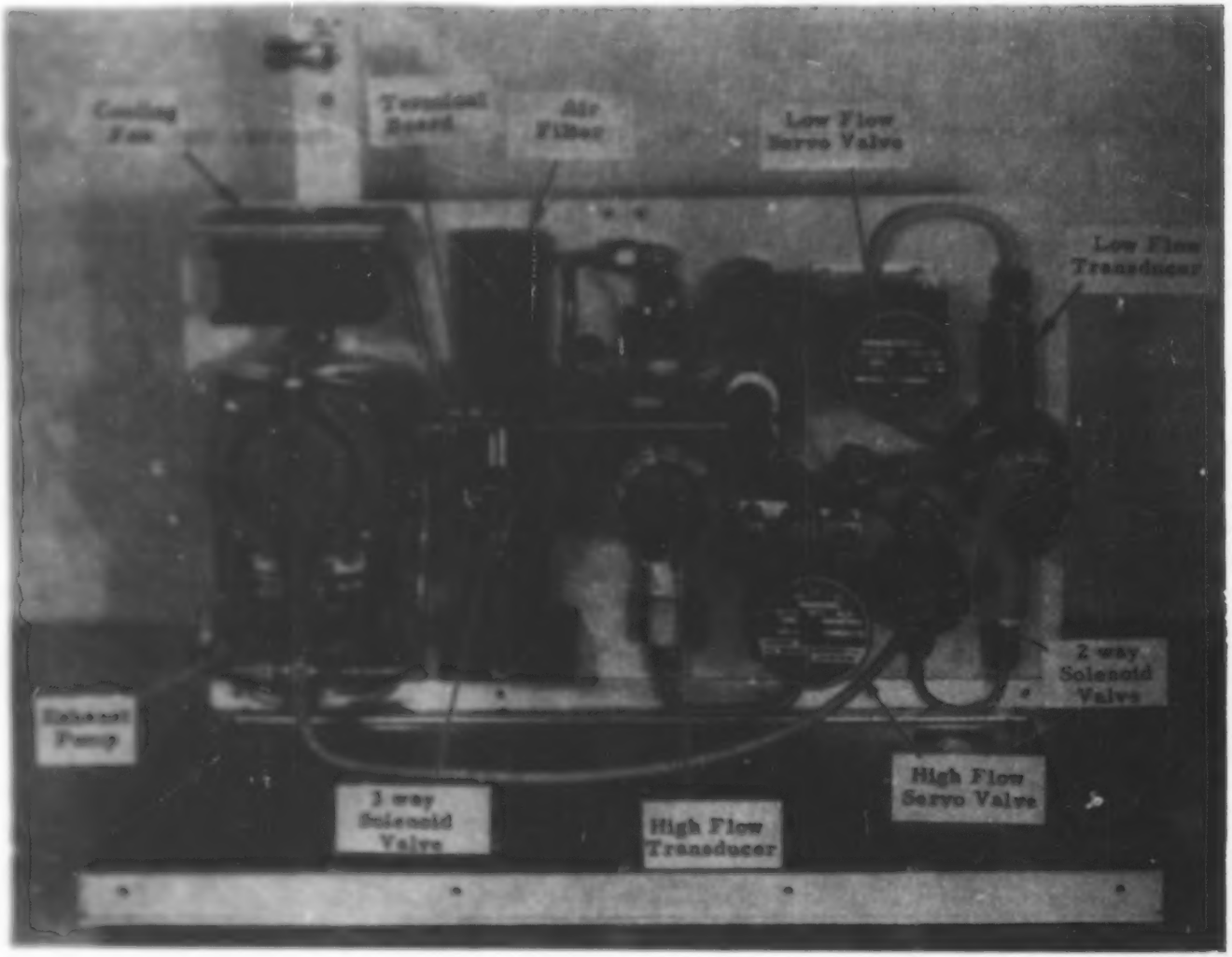

Figure A-15. Inside View, Diluter Module-Front Section Removed

acetone must then be removed by pumping air through the transducer for about 15 minutes. The transducer may then be reinstalled and used immediately.

The Pump Module

The Pressure regulator is the maintenance concern of the pump module. If a filter is used on the air intake of the regulator, it is only necessary to change the filter when it gets dirty. However, if no filter is used and the regulator gets dirt inside, it is then necessary to take the regulator apart and clean it. The indication that the regulator is dirty is that the regulator adjustment has no effect on the pressure reading of the magnehelic gauge.

Again, disconnect the power, first. Then the regulator is removed by disconnecting the gas line and removing the two screws on the bottom of the unit. A vise is needed to hold the regulator while the top is removed with a wrench. Care must be taken when doing this as there are two springs inside. Once apart the orifice is cleaned, and the regulator is then put back together and replaced in the pump unit.

APPENDIX B

SAMPLING PROCEDURES

For the field test, comparison of the instrumental technique with a simple bubbler collection and Hatch and Ott analytical technique was carried out.

The wet chemical technique utilized standard midget impingers which were connected by glass ball and socket joints. The first bubbler contained 15 ml of saturated sodium bicarbonate which served to remove sulfur dioxide from the gas stream. The second contained 1.0 ml of 0.25 M potassium permanganate and 2.5 ml of 1:1 nitric acid diluted to 15 ml. The sodium bicarbonate impinger was used because of sulfur dioxide interference.

A vacuum pump and flowmeter were used to obtain a flow rate of 2.0 liters per minute through the impingers. They were connected to the glass tee at the end of the probe and then removed after 2.5 minutes. A sampling time of 2.5 minutes was found to be the optimum with the amount of potassium permanganate used. After taking a duplicate sample, the samples were analyzed.

With the instrumental methods, a check was made to see if the proper operating conditions existed. The gas flows were set at 1.5 l/min through the photometer and 0.5 l/min through the waste line. A visual check of the scrubber was sufficient to determine that the scrubber fluid flow was appropriate. The temperature of the probe was maintained approximately at the duct temperature (280°-320°F) while the Gelman filter holders were kept warm to the touch. New filter paper was put in the filter holders every 2 hours.

With the recorder on the 2 millivolt scale and a chart speed of 0.5 inches per minute, a signal was obtained from the stack gas. The three-way valve was then turned directing the gas through the 10% silver-alumina tablets. With the mercury removed, the gas was directed through the photometer again. After about one minute, the three-way valve was returned to the original position. The quantity of mercury represented by the difference of the two signals was then determined. From a mercury-containing flask, 1.0 to 2.0 cubic centimeters of mercury vapor was drawn into a syringe and injected into the sample line near the probe. With a chart speed of 1 inch per second, a large peak was obtained which was used to quantitate the unknown stack gas mercury level (if the peak was too large for the 2 millivolt recorder scale, the 5 millivolt scale was used). The amount of mercury was determined by injecting a similar amount into a midget impinger containing the same acid permanganate solution

as used in the previous procedure.* The contents of the impinger were then analyzed.

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* Normally the amount of mercury could be determined by the vapor temperature, however, during the test the thermometer available (19 -27) was off scale most of the time.

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* Normally the amount of mercury could be determined by the vapor temperature, however, during the test the thermometer available (19 -27) was off scale most of the time.

ANALYTICAL PROCEDURE

A modified Hatch and Ott procedure was used to determine the mercury content of the various impingers. To avoid possible interferences, the contents of the impingers were reduced with 3.0 milliliters of 20 percent weight/volume stannous chloride in 6 normal hydrochloric acid. The released mercury was recaptured in a midget impinger containing 0.3 ml of 0.25 molar potassium permanganate and 2.5 ml of 1:1 nitric acid diluted to 15 ml. With a vacuum pump, air was pulled through the coupled impingers at 2.0 liters per minute for 3 minutes.

The photometer section of a GEOMET Model 103 Mercury Monitor was then prepared to read the peak height obtained from reducing this impinger. An empty midget impinger was connected to the photometer intake line which has an air flow of 2.0 1/min. To the empty impinger, another impinger was connected containing 1 gram of sodium borohydride in 15 ml of water. The contents of the mercury-containing impinger was then reduced with 2.0 ml of the stannous chloride solution. After 10 seconds of vigorous shaking, it was hooked to the sodium boroyhydride impinger, and the peak height measurement was given by the differential voltmeter of the mercury monitor. The amount of mercury present in the impingers was determined from the calibration curve of the mercury monitor.